Daniela Patricia Alves Mainhas

Bachelor Degree in Chemical and Biochemical Engineering

Start-up and optimization of pilot and industrial scale membrane systems for pre-concentration of microalgae

Dissertation to obtain Master Degree in Chemical and Biochemical Engineering

Orientador: Doutor Edgar Santos, Diretor de Projetos Industriais, A4F – Algafuel S.A.

Júri:

Presidente: Doutor João Paulo Serejo Goulão Crespo, Professor Catedrático, FCT NOVA

Arguente: Doutora Carla Maria Carvalho Gil Brazinha de Barros Ferreira, Investigadora, FCT NOVA

Supervisor: Doutor Edgar Tavares dos Santos, Diretor de Projetos Industriais, A4F – Algafuel S.A.

FACULDADE DE
CIÊNCIAS E TECNOLOGIA
UNIVERSIDADE NOVA DE LISBOA

November, 2020

Daniela Patricia Alves Mainhas

Bachelor Degree in Chemical and Biochemical Engineering

Start-up optimization of pilot and industrial scale membrane systems for pre-concentration of microalgae

Dissertation to obtain Master Degree in Chemical and Biochemical Engineering

Orientador: Edgar Santos, Diretor de Projetos Industriais, A4F – Algafuel S.A.

Jury:

Presidente: Doutor João Paulo Serejo Goulão Crespo, Professor Catedrático, FCT NOVA

Arguente: Doutora Carla Maria Carvalho Gil Brazinha de Barros Ferreira, Investigadora, FCT NOVA

Supervisor: Doutor Edgar Tavares dos Santos, Diretor de Projetos Industriais, A4F – Algafuel S.A.

Faculdade de Ciências e Tecnologias

Universidade Nova de Lisboa

November, 2020

Start-up optimization of pilot and industrial scale membrane systems for pre-concentration of microalgae

Copyright © Daniela Patricia Alves Mainhas, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa.

A Faculdade de Ciências e Tecnologia e a Universidade Nova de Lisboa têm o direito, perpétuo e sem limites geográficos, de arquivar e publicar esta dissertação através de exemplares impressos reproduzidos em papel ou de forma digital, ou por qualquer outro meio conhecido ou que venha a ser inventado, e de a divulgar através de repositórios científicos e de admitir a sua cópia e distribuição com objetivos educacionais ou de investigação, não comerciais, desde que seja dado crédito ao autor e editor.

Acknowledgements

First, I want to start by thanking A4F and all its representatives for welcoming me and always being so helpful whenever I needed. I want to especially thank Edgar Santos and Luis Costa for helping me with my thesis and for their availability and Rui Mota for helping me with the basis for my thesis. Also want to thank Margarida Duarte and Sara Pereira for the advices and all the people working in the unit for always being available to help me.

I also want to thank my parents for making this possible and always being there to support me, my friends for all the exchanged ideas and support and my boyfriend for is unconditional support.

Abstract

Microalgae is an industry that has been evolving a lot throughout the later years and has quite some advantages especially to the environment, from the consumption of carbon dioxide to the replacement of fossil fuel with biofuel. But, in order to produce microalgae, they need a medium to grow that needs, later, to be separated in order to obtain only the microalgae.

There are several ways to separate the algae from the medium, among which are centrifugation, flocculation and sedimentation, but they have their disadvantages as they are time-consuming, uneconomic and bad for the environment. On the other hand, there is the filtration that has more advantages as being able to recover all the processed biomass, having a limited addition of chemicals and it has a low energy consumption.

On this case an ultrafiltration system will be optimized for the microalgae to be harvested and passed through this system to separate them from the medium. The problem with this system is that it is a very low-pressure system, making it very hard to clean after the filtrations. The goal is to find an efficient way to clean it that is less time consuming and that allows the system to be ready to use every time an harvest is due.

Keywords: Microalgae, centrifugation, flocculation, sedimentation, filtration, ultrafiltration.

Resumo

A indústria das microalgas tem vindo a evoluir bastante nos últimos anos, o que tem algumas vantagens especialmente para o meio ambiente, desde o consumo de dióxido de carbono para a possível substituição de combustíveis fósseis por biocombustíveis. Mas, para produzir as microalgas, é necessário um meio para as mesmas crescerem e, mais tarde, é necessário separar as microalgas desse meio.

Existem várias maneiras de separar as microalgas do meio, entre elas a centrifugação, floculação e sedimentação, mas têm as suas desvantagens pois são demoradas, não são económicas e não são ecológicas. Por outro lado, existe a filtração que tem as suas vantagens, é capaz de recuperar toda a biomassa, tem muito pouca adição de químicos e baixo consumo de energia.

Neste caso, um sistema de ultrafiltração será otimizado de forma a que as microalgas sejam colhidas e passem por este mesmo sistema para serem separadas do meio. O problema com este sistema é funcionar com baixas pressões, o que faz com que seja bastante difícil de limpar. O objetivo é encontrar uma maneira de o limpar que seja eficiente e que não consuma muito tempo, de forma a que o sistema esteja pronto a ser usado cada vez que for necessário para uma colheita.

Palavras-chave: Microalgas, centrifugação, floculação, sedimentação, filtração, ultrafiltração.

List of contents

List of figures

List of tables

List of abbreviations

ρ – Density.

A4F – Algae for the future.

BC – “Production” program with recurrent backwashes.
BL – Air blower.
BW – Backwash with the pump at 50 Hz.

C – Concentration.
$C_6H_7O_7^-$ – Dihydrogen isocitrate.
$C_6H_8O_7$ – Citric acid.
CA – Cellulose acetate.
CAC – Cleaning with citric acid.
CAS – Solution with citric acid.
CF – Cleaning the filters.
CIP – Cleaning in place.
CS – Solution of sodium hypochlorite.
CSC – Cleaning with a solution of sodium hydroxide and sodium hypochlorite.
CSS – Solution with sodium hydroxide.

DP – Dose pump.

F – Filtration.
FI – Flow indicator.
FT – Flow transmitter.

H_2O – Water.
H_3O^+ – Hydronium.
HMWC – High molecular weight component.

Ka – Acid dissociation constant.

LMWC – Light molecular weight component.
LS – Level sensor.

m – Weight.

M – Molecular weight.

MB – Manual backwash cleaning.

n – Mol.

NaClO – Sodium hypochlorite.

NaOH – Sodium hydroxide.

NRV – No return valve.

OH^- – Hydroxide.

P – Pump.

P&ID – Piping and instrument diagram.

PC – Permeability Check.

PI – Pressure indicator.

PS (PSO) – Polysulfone.

PT – Pressure transmitter.

PVDF – Polyvinylidenedifluoride.

T – Tank.

TMP – Transmembrane pressure.

V – Volume.

VA – Valve.

VFD – Variable frequency drive.

VM – Manual valve.

WH – Washing with a hose.

1 Thesis goal

The goal of this thesis is to optimize the harvest filtration of an ultrafiltration system and its cleaning process.

Through this master thesis I will be working with three different microalgae, *Dunaliella sp.*, *Nannochloropsis sp.* and *Odontella sp*.

The first step will be to get familiar with the operation system and understand how the machine records its data in order to analyze it.

It is important to gather all the information from all the filtrations and cleaning steps that were done before this master thesis and analyze it, to see what can be done as cleaning steps and what are the most important parameters to analyze, when performing a harvest filtration.

After this, the cleaning steps should start to be performed to see which steps are the most efficient ones and all the important harvest parameter should be changed, until the optimal parameters for the filtration of each algae is found.

It is also important to consider all the system flaws and figure a way to try and solve them.

2 Introduction

2.1 Company

A4F is a Portuguese biotechnology company specialized in microalgae. It researches and produces microalgae and it designs, builds, operates and transfers commercial-scale microalgae production units.

A4F has its headquarters in Lisbon, were the research is done and the prototypes are produced for scaling up into larger facilities.

In 1989, some members of A4F management team, in the biotechnology superior school of the Catholic University of Porto, started investigating microalgae biotechnology. Then, in 1997, in Olhão, the founders of A4F started producing and selling microalgae. After nine years, in 2006, A4F was finally founded and started its first project one year later, in 2007, with a cement company called SECIL. The project had the objective of reducing SECIL's carbon footprint.

In 2009, a P&ID unit was founded in Pataias, called Algafarm, with the capacity of more than 1.300 m^3 of production. Its construction started in 2010 and was finished in 2012. The project was finished in 2015 and was then delivered to the clients.

A4F has two implemented industrial projects, Algafarm and Biofat, both in Pataias, and two projects being implemented, Biofabrica in Aveiro and Algatec in Lisbon.

2.2 Microalgae

Microalgae's industry has increased a lot over the last decades [1], since in the early 1950's the increase of the world's population and predictions of an insufficient protein supply led to a search for new alternatives of protein sources [2].

Microalgae are microscopic single cells that can grow in different aquatic habitats, such as lakes, rivers, oceans and wastewaters [1]. These organisms grow really fast [3] and can tolerate different ranges of temperature, salinities, pH values and light intensities [1]. They are rich sources of carbon [1] and have different ways to fix atmospheric carbon dioxide to use it to convert into biomass [3] that can be used in human food, food supplements, biofuels, bio products, pharmaceuticals and cosmetics [1].

Dunaliella sp. is a microalgae that accumulates carotenoids, under stress conditions, and has various applications in health and nutritional products. It is also characterized by its lack of a rigid wall, making it harder to filter [4].

Nannochloropsis sp. can tolerate broad environmental and culture conditions and has a high industrial demand due to its rapid grow, ability of synthesizing large amounts of triacylglycerols and high value polyunsaturated fatty acids [5].

Odontella sp. can be used as a dietary supplement rich in ω-3 polyunsaturated fatty acids. It also has some bioactive compounds that can be used to benefit human health, such as fucoxanthin and phytosterols [6].

2.3 Microalgae harvesting

There are various methods used for microalgae harvesting, such as centrifugation, flocculation, flotation and sedimentation. The advantages and disadvantages of each method can be seen in Table 2.1. [7][8]

Table 2.1 – Advantages and disadvantages of the different harvesting methods. [9][10]

Microalgae Harvesting	Advantages	Disadvantages
Centrifugation	-High cell recovery -Applicable to many species -Fast and efficient	-Uses a lot of energy -Cell damage -High cost
Flocculation	-Low cost -Applicable to many species -Less cell damage	-Uses chemicals -Hard to separate the coagulant -pH dependent
Flotation	-Short operation time -Low cost - Suitable for unicellular microalgae	-Contamination -Needs surfactants
Sedimentation	-Low cost -Energy efficient -Can separate larger microalgae	-Slow rate -Cannot be used for smaller microalgae

An alternative to these methods is filtration. This method consists in a pressure drop across the filter membrane to make the microalgae cultures pass through a filter membrane where microalgae get retained on the surface and the water flows through the membrane [11]. This method has many advantages such as being able to recover all the processed biomass, has a limited addition of chemicals and it has a low energy consumption [7][12]. The most important disadvantage of filtration is the membrane fouling, that reduces the membrane life time and the permeate flow, and increases the energy consumption [7].

Centrifugation is the most used method for harvesting microalgae but, when it comes to smaller microalgae and lower concentrations, there is a need for an increased velocity in centrifugation which will damaged the microalgae and let its contents flow out [13].

On the other hand filtration is commonly used for microalgae harvesting due to the possibility of rejecting all algae cells and low energy waste [14].

2.3.1 Filtration

Filtration using pressure, such as microfiltration and ultrafiltration, are the most used membrane technology processes, due to its hight efficiency and easy operation and scalability [15], but, it has a big disadvantage that is the fouling caused by the microalgae cells that get attached to the surface of the membrane, which will be the main problem to solve throughout this master thesis [13].

There are four types of membrane filtration according to their size exclusion: reverse osmosis, nanofiltration, ultrafiltration and microfiltration. The differences between them are described in Table 2.2 [16][17].

Table 2.2 - Different types of membrane filtration [16].

	Reverse Osmosis	Nanofiltration	Ultrafiltration	Micro filtration
Membrane	Asymmetrical	Asymmetrical	Asymmetrical	Symmetrical Asymmetrical
Thickness Thin film	150 µm 1 µm	150 µm 1 µm	150 - 250 µm 1 µm	10 - 150 µm -
Pore size	<0.002 µm	<0.002 µm	0.2 - 0.002 µm	4 - 0.002 µm
Rejection of	HMWC, LMWC sodium chloride glucose amino acids	HMWC mono-, di- and oligosaccharides polyvalent neg. ions	Macro molecules, proteins, polysaccharides vira	Particles, clay bacteria
Membrane material(s)	CA Thin film	CA Thin film	Ceramic PSO, PVDF, CA Thin film	Ceramic PSO, PVDF
Membrane Module	Tubular, spiral wound, plate-and-frame	Tubular, spiral wound, plate-and-frame	Tubular, hollow fiber, spiral wound, plate-and-frame	Tubular, hollow fiber
Operating pressure	15 - 150 bar	5 - 35 bar	1 - 10 bar	<2 bar

2.3.1.1 *Ultrafiltration*

Ultrafiltration membranes are anisotropic structures that uses a finely porous membrane to separate water and microsolutes from macromolecules and colloids [18].

The molecular weight of the solute is the most important factor to affect the permeation of the membrane, but there are several other factors. One of these factors is the shape of the molecules being retained. If there are two molecules, for example polydextran and a protein of the same molecular weight, polydextran will have a lower rejection than the protein. This happens because polydextran is a linear molecule and can more easily sneak through the membrane pores. On the other hand, proteins are tightly wound globular coils that are held together by hydrogen bonds, so it is impossible for the proteins to deform themselves in order to pass through the membrane pores [18].

Another factor that influences the permeation of the membrane is the pH of the solution. molecules can behave differently at different pH. For example, polyacrylic acid at a pH equal or higher than 5 is very extended and relatively inflexible so it cannot pass through the membrane, but at a pH equal or lower than 3 the neutral molecule is much more flexible and can pass through the membrane pores [18].

2.3.1.2 *Cross-flow and in line filtration*

Membrane filtration can be done in two different ways, cross flow or in line filtration, these two ways occurred as described in Figure 2.1. The cross-flow filtration has advantages when compared to the conventional filtration (in line). The main advantage is that it limits the formation of cake since the majority of this is washed away during filtration, which is a way of extending the membrane life time, since the higher the cross flow velocity the more difficult is for the biomass to accumulate on the membrane. Studies show that this type of filtration is the most energy efficient method for harvesting microalgae [7][19].

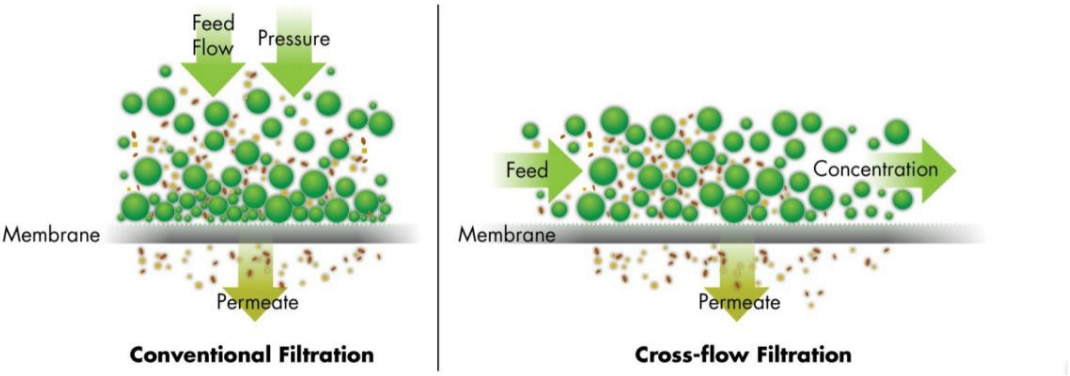

Figure 2.1 – Cross-flow filtration versus in line filtration [19].

2.3.1.3 Outside-in and inside-out filtration

Outside-in filtration is when the culture is feed outside of the membranes and it is sucked into the membranes in order to filtrate. Inside-out filtration is when the culture is fed into the membranes and is pushed to the outside of the membrane in order to filtrate. In this case the system works as a outside-in filtration [20].

2.3.1.4 Membrane fouling

Membrane fouling is usually caused by molecules or particles that can precipitate or deposit onto the membrane surface or pores. This fouling will make it harder to separate the intended components, reduce its productivity and alter the membrane selectivity [21].

The solution that permeates the membrane drags solvent and macromolecular or colloidal solutes to the surface of the membrane. The solvent and the small solutes permeate the membrane but the larger solutes accumulate at the surface of the membrane due to their large size. While filtrating, the solutes will become more and more concentrated forming a gel layer that will work as a secondary barrier to the flow passing through the membrane (Figure 2.2). This layer formation can be mathematically described by Equation 1 [18].

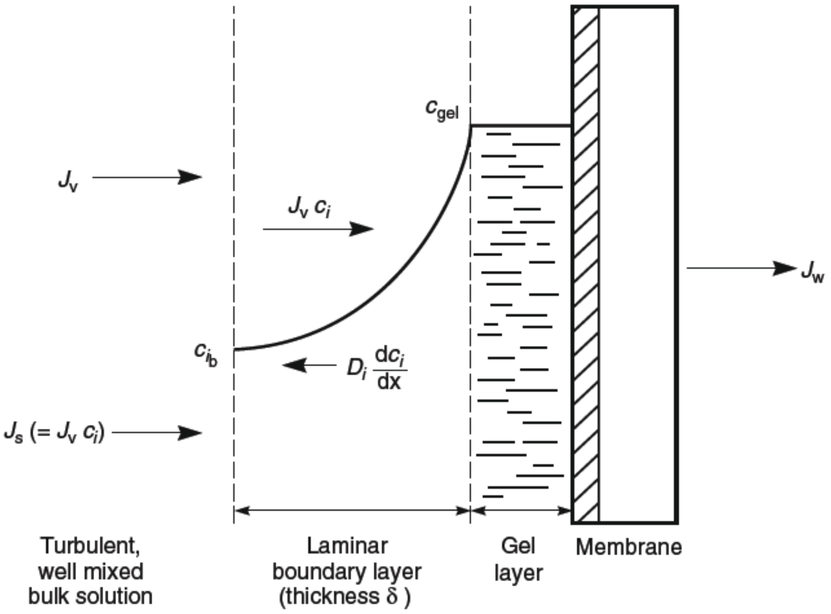

Figure 2.2 – Formation of a gel layer onto the surface of the membrane [18].

$$J_v c_i = D_i \frac{dc_i}{dx}$$

Equation 1 [18]

J_v – Volume flux;

c_i – Solute concentration;

D_i – Diffusion coefficient of the macromolecules in the boundary layer.

There are three major factors that affect the membrane fouling. The first one is the material from which the membrane is made, such as its morphological structure and surface properties. The second one is the properties of what is filtrated using the membrane, such as composition, concentration, pH and ionic condition. The third and final one is the operation conditions in which the filtration with the membrane occurs, TMP, cross-flow velocity, temperature, backwashing, among others [21].

There are four types of fouling, inorganic fouling, particulate/colloid fouling, microbial/biological fouling and organic fouling [22].

The inorganic fouling is caused by the accumulation of inorganic precipitates such as crystallized salts, oxides and hydroxides. This type of fouling does not occurs as much in the microfiltration and ultrafiltration as it occurs in other types of filtration [22][23].

The particulate/colloid fouling happens when suspended solids and/or colloidal material, such as algae, bacteria and certain natural organic matters, clog the holes of the membrane or adhere to its surface [22][23].

The microbial/biological fouling is when there is a formation of biofilms on the membrane surface. For example when plants, algae or bacteria start to grow and form an extracellular polymetric substance [22][23].

The organic fouling is caused by the collection of carbon-based material on the membrane. This compounds can be commonly found in soil, ground and surface water like rivers or lakes [22][23].

In order to prevent as much fouling caused by the microalgae techniques have been developed to prevent as much fouling caused by the algae. This techniques are air assisting the backwash, preozonation of the suspension, and chemical treatment of the fouled membrane [20].

2.3.1.5 Membrane cleaning

Caustic or alkaline cleaners are effective for many organic foulants and some inorganic foulants. The alkaline cleaning can saponificate fats and lipids, making them soluble in water, neutralizing humic acids and dispersing colloidal material [21]. Sodium hydroxide (caustic soda) promotes the dissolution of weakly acidic organic matter and cleavage of polysaccharides and proteins into smaller sugars and amides. It also expands natural organic matter molecules transferring the cleaning agent solution to the membrane surface [24]. Examples of this cleaners are sodium hydroxide, soda ash, phosphates, hypochlorite and potassium hydroxide.

Acidic cleaners are used to clean scale compounds and metal oxides by making them soluble and chelating. They can also be used to dissolve precipitates that are formed during the alkaline cleaning. Examples of this cleaners are nitric acid, hydrochloric acid, phosphoric acid and citric acid [21]. Citric acid is a good option due to its good chelating abilities and being easy to use with a lower risk of pH damage when compared to mineral acids [24].

Oxidants are able to degrade the foulant by increasing the number of functional groups that contain oxygen and make it more susceptible to hydrolysis at high pH levels. Examples of oxidants are hydrogen peroxide and sodium hypochlorite, being the sodium hypochlorite the most used one [21][24].

Sanitizers and disinfectants are used after every cleaning to store the membrane between uses. Oxidants can be used for this effect [21].

3 Materials and Methods

3.1 Filtration system

The filtration unit is an ultrafiltration system design for the filtration of algae. It can be used as a pre-concentrator for centrifugation or for water recycling coming from the centrifuge.

3.1.1 Operation of the filtration system

In Figure 3.1 the P&ID.

The filtration unit has a manual program and three automatic programs: “Production”, “CIP” and “Fill T-01”. The manual program allows us to control every equipment that is in the P&ID.

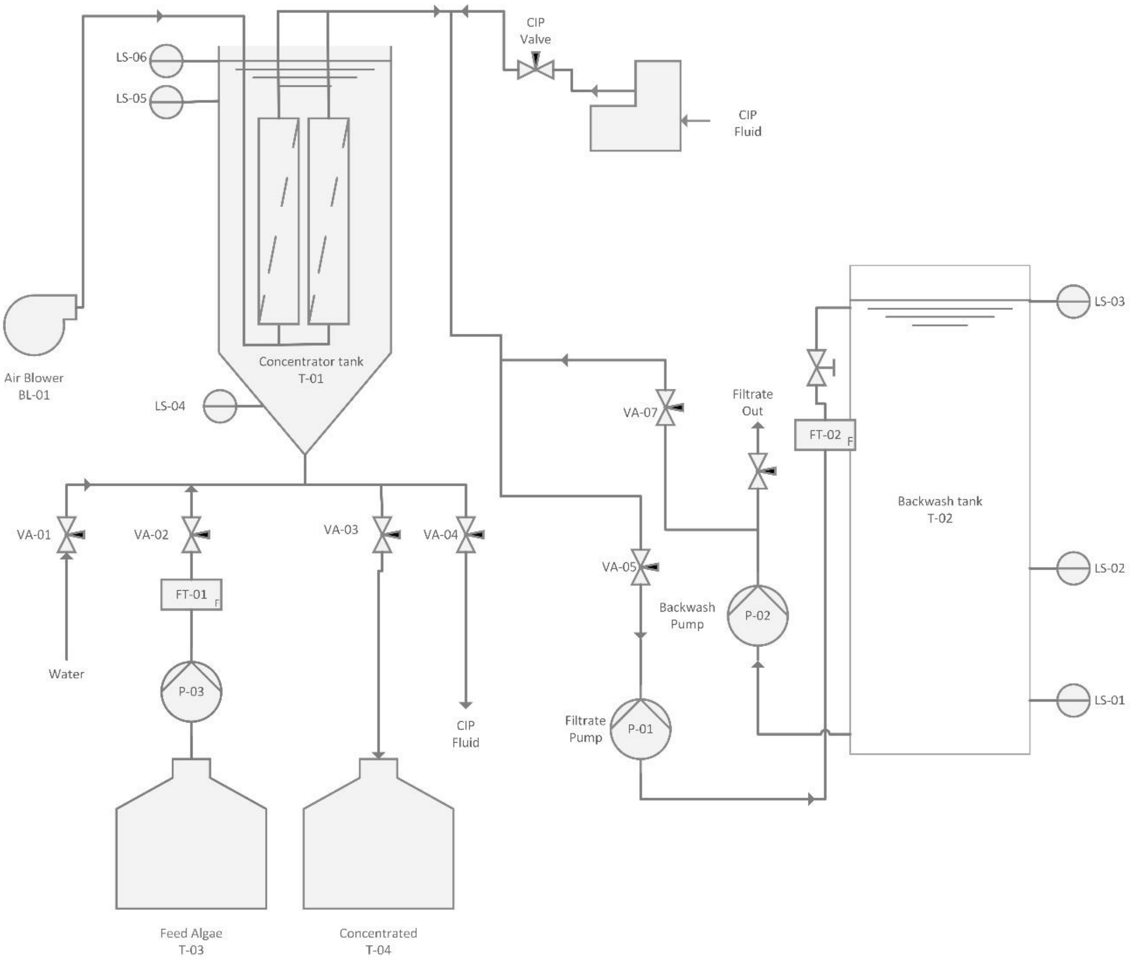

Figure 3.1 – Filtration unit P&ID.

The “Production” automatic program is used to concentrate the algae. For this program we have to choose the settings that are: “Process volume”, “Cycle counter”, “Backwash interval time”, “Backwash duration”, “Harvest delay”, “TMP”, Backwash speed”, “Low filtrate alarm” and “Delay filtrate”.

The “Production” program starts when the valve VA-02 opens and the feed pump (P-03) starts working, the algae culture is fed into the concentrator tank (T-01). When it reaches the sensor LS-05 the valve VA-05 opens and the filtrate pump (P-01) starts working to fill the permeate tank (T-02). If the permeate flow is lower that the feed flow and the concentrator tank level reaches the sensor LS-06, the feed pump will stop until the level of the concentrator tank goes bellow the sensor LS-05, and then turn back on again. If the level of the permeate tank reaches the sensor LS-03, the valve VA-06 opens and the backwash pump (P-02) starts to remove the permeate until the level on the permeate tank goes below the sensor LS-02. The backwash interval time is the time since permeate flow starts until the filtration system performs a backwash and between backwashes. So according to the time chosen in the settings, the filtrate pump will stop and the valve VA-05 will close, and right after, the valve VA-07 will open and the backwash pump will start working, during the backwash duration chosen in the settings. After that time passes the backwash pump will stop and the valve VA-07 will close so that the VA-05 opens and the filtrate pump starts working. This allows the biomass that accumulates on the membrane to come off and the permeate flow to increase. The FT-01 measures the feed flow and with that the filtration system calculates the volume that has been processed. When that volume reaches the process volume chosen on the settings, the feed pump will stop and the VA-02 will close. Then the unit will continue to filtrate until the level on the concentrator tank goes bellow the sensor LS-05 and the filtrate pump and valve VA-05 will stop, leaving the concentrator tank with 466L of concentrated microalgae culture. After the “Production” program we can remove the SD card and analyze the data (values from 10 to 10 seconds of feed flow, permeate flow and TMP).

The “CIP” automatic program is used to clean the unit after a filtration. For this program we have to choose the settings that are: “CIP soak time” and “CIP dose time”.

The “CIP” program starts when valve VA-01 opens and the concentrator tank starts being filled with water. When the concentrator tank level reaches the sensor LS-05 the valve VA-01 closes, the air blower (BL-01) turns on and the CIP valve opens during the time chosen on the “CIP dose time”. After the time chosen on the settings for the “CIP soak time”, the blower stops and the “CIP” program is concluded.

The “Fill T-01” automatic program is used to fill the concentrator tank.

The “Fill T-01” program starts when valve VA-01 opens and the concentrator tank starts being filled with water. When the concentrator tank level reaches the sensor LS-05 the valve VA-01 closes and the tank stays filled with water.

3.2 Membrane Area

Each membrane module has 4 bundles of membrane fibers, each bundle with 12 lines of 60 fibers each. The membrane fibers have a height of 1.48 m and 2 mm of diameter. Knowing this, the membrane area of filtration can be calculated through the Equation 2, Equation 3 and Equation 4. Since there are two membranes with the same area in the system, the total membrane area is calculated through Equation 5.

$$Amount\ of\ membrane\ fibers = 60 \times 12 \times 4 = 2880$$

Equation 2

$$Area\ of\ each\ membrane\ fiber = 2\pi \times \frac{0{,}002}{2} \times 1{,}48 = 0{,}0093\ m^2$$

Equation 3

$$Membrane\ area\ of\ filtration = 0{,}0093 \times 2880 = 26{,}78\ m^2$$

Equation 4

$$Total\ membrane\ area\ of\ filtration = 26{,}78 \times 2 = 53{,}56\ m^2$$

Equation 5

Figure 3.2 – Membrane.

3.3 Permeability

3.3.1 Permeability checks

In order to know what the permeability of the membrane is, a permeability check must be performed. These tests are done by performing a “Production” program on the system, with water and without any backwashes (Figure 3.3), or doing it manually, opening up the water valve (VA-01) to fill the concentrator tank with water, until the sensor LS-06 turns on. Then, with the blower turned on, the valve VA-05 must be open and the pump P-01 turned on. If the level on the permeate tank reaches the sensor LS-03 then the valve VA-06 must be opened and the pump P-02 turned on until the sensor LS-02 turns off. When the sensor LS-05 turns off, all pumps must be stopped and valves closed (Figure 3.4).

After the permeability check is performed, the SD card is removed to gather the recorded results in order to do a graphic of the feeding flow and permeate flow in function of the time (Figure 3.3 and Figure 3.4), and of the TMP in function of the time (Figure 3.5). The goal is to restore the permeability to at least 90% of the original permeability of the membrane. If this is not accomplished some more cleaning steps are needed.

The TMP graphics do not need to be analyzed in the permeability checks because the TMP is not changed during it, so the TMP always goes accordingly to the permeate flow (Figure 3.4 and Figure 3.5).

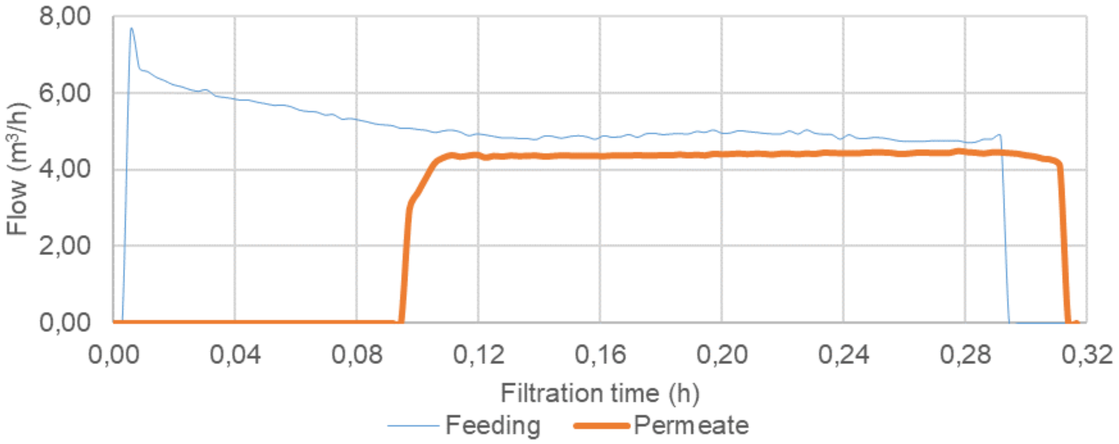

Figure 3.3 – Permeability check results using the "production" program.

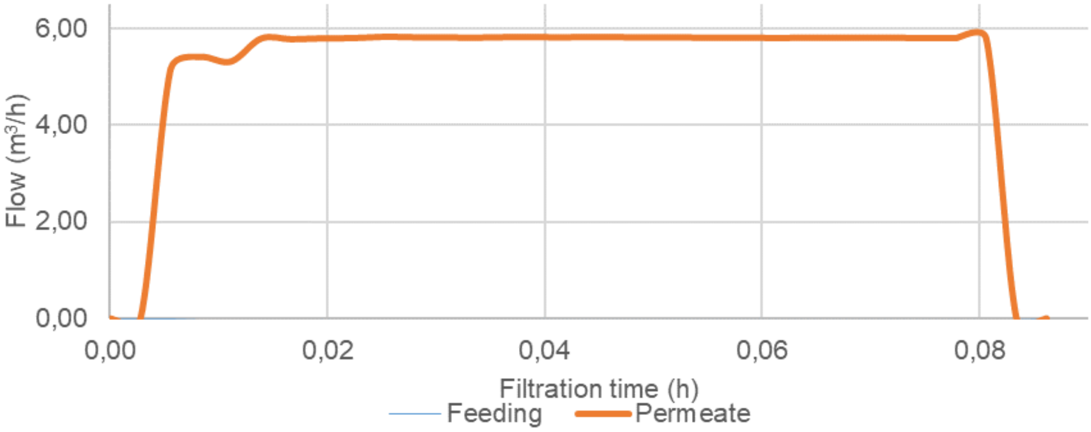

Figure 3.4 – Results of a manual permeability check.

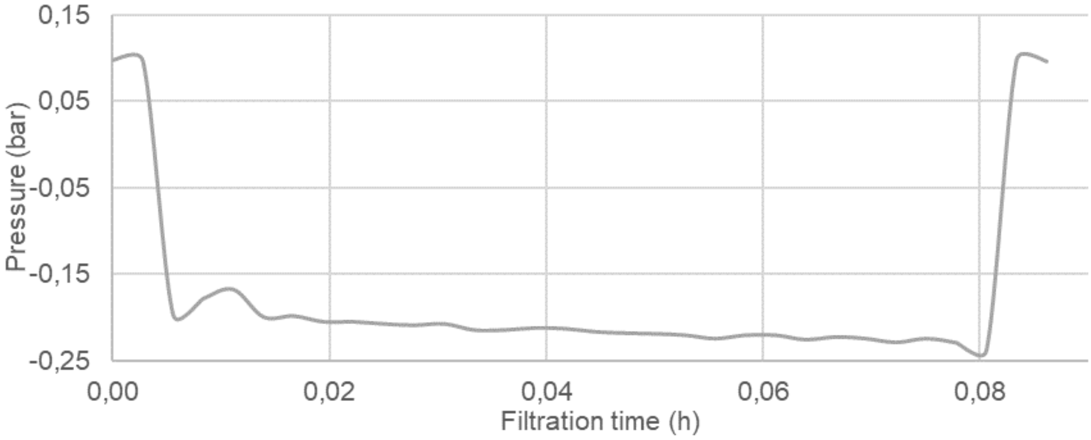

Figure 3.5 – TMP in function of the time for the permeability check on Figure 3.4.

3.3.2 Membrane permeability

When the system first arrived, a permeability check to see the maximum value of permeability of the membrane was not performed, and this information was not available from the supplier. Therefore, the maximum permeability will have to be considered as the highest value obtained in all of the permeability checks done.

The membrane permeability can be calculated using Equation 6.

$$Permeability\ of\ the\ membrane\ \left(\frac{L}{m^2.h.bar}\right) = \frac{Permeate\ flow\ (\frac{L}{h})}{Membrane\ area\ (m^2) \times TMP(bar)}$$

Equation 6 [25]

The SD card gives values of permeate flow and TMP from 10 to 10 seconds, with these values the permeability of the membrane is calculated also from 10 to 10 seconds and then the average permeability is done in order to obtain the permeability of the membrane during that permeability check.

The recovery of the membrane is the percentage of permeability obtained in a permeability check in comparison to the highest value of permeability ever obtained. It can be calculated throughout Equation 7.

$$Recovery\ of\ the\ membrane\ (\%) = \frac{Permeability\ of\ the\ membrane\ \left(\frac{L}{m^2.h.bar}\right)}{Maximum\ permeability\ of\ the\ membrane\ \left(\frac{L}{m^2.h.bar}\right)}$$

Equation 7

3.4 Cleaning optimization

After every filtration the membrane should be cleaned up in order to restore its permeability. There are two types of cleaning, using just water by backwashing and forward washing or using chemicals. The use of chemical is needed when the permeability of the membrane can no longer be restored to a sufficient value just with water cleaning, but it is a cleaning then when is used very often, also causes fouling of the membrane[26].

Depending on the type of fouling on the membrane, there are two types of chemical cleaning, acid cleaning and basic cleaning. The basic cleaning is the most used one because it is used to cleaning organic matter, and then the acid cleaning is used to clean the inorganic matter, so is not so it is not used as often [26].

3.4.1 Cleaning Steps Description and Results Legend

- **Manual backwash cleaning (MB):** Having the concentrator tank full and the manual program on, we open the valve VA-05 and turn on the filtrate pump, when the sensor LS-05 turns off we turn off the filtrate pump and close the VA-05 valve. Then we open the VA-07 valve and turn on the backwash pump until the LS-06 sensor turns on, when this happens we turn off the backwash pump and close the VA-07 valve. We repeat this procedure until the permeate flow rate remains constant.

- **Backwash with the pump at 50 Hz (BW):** With a hose the permeate tank is filled with water, then having the manual program on, the VA-04 and VA-07 valves are opened and the backwash pump is turn on until the sensor LS-01 turns off.

- **“Production” program with recurrent backwashes (aBCb):** It is a “Production” program to filtrate a solution of 50 ppm of sodium hypochlorite with recurrent backwashes, “a” corresponds to the backwash interval time and “b” corresponds to the backwash duration.

- **Washing with a hose (WH):** Washing the concentrator tank from the top with water using a hose.

- **Cleaning the filters (CF):** Removing the pump filters, washing them and leaving them in a solution of 50 ppm of sodium hypochlorite overnight.

- **Solution of citric acid (aCAS):** This corresponds to the number of days that the membrane was left inside a solution of 0.05 M of citric acid, “a” corresponds to the number of days that the membrane stayed inside this solution.

- **Solution of sodium hydroxide (aCSS):** This corresponds to the number of days that the membrane was left inside a solution of 0.1 M of sodium hydroxide, “a” corresponds to the number of days that the membrane stayed inside this solution.

- **Solution of sodium hypochlorite (aCS):** This corresponds to the number of days that the membrane was left inside a solution of 50 ppm of sodium hypochlorite, “a” corresponds to the number of days that the membrane stayed inside this solution.

- **Permeability check (PC)**

- **Filtration (F)**

- **Cleaning in place (aCIP):** “a” corresponds to the CIP soak time.

- **Cleaning with citric acid (aCACb):** It is a “production” program with a 0.02 M solution of citric acid using 2000 L of solution that is kept recirculating, “a” corresponds to the minutes that the system was performing a cleaning and “b” corresponds to the interval in-between backwash.

- **Cleaning with a solution of sodium hydroxide and sodium hypochlorite (aCSCb):** It is a “production” program with a solution of 0.04% of sodium hydroxide and 200 ppm of sodium hypochlorite using 2000 L of solution that is kept recirculating, “a” corresponds to the minutes that the system was performing a cleaning and “b” corresponds to the interval in-between backwash.

3.4.2 Cleaning with a solution of sodium hydroxide and sodium hypochlorite

The cleaning will be performed using the “production” program and the settings will be chosen according to the membrane cleaning needs. Since the system does not allow a time to be set for the duration of a “production” program, than the volume will have to be set for its highest (100 m^3) and the program will have to be stopped when the intended time passes. For the basic cleaning the time should be between 30 and 240 minutes and the backwash interval of time should be between 1 and 3 minutes[27]. This time will be chosen according to the fouling of the membrane. All the other settings will be maintained as the usual values because it will not change anything in the cleaning process.

The harvest tank will be filled with 2000 L of solution in order to keep recirculate the solution during the time needed.

The information from the supplier regarding the cleaning of the membrane with a basic solution is that it should be used a mixed solution of 0.04% of sodium hydroxide and 200 ppm of sodium hypochlorite with a pH equal or lower than 12.

The sodium hydroxide solution existing in the operation site is a 32% solution and the sodium hypochlorite solution is a 13%.

- For 0.04% of NaOH:

[NaOH]= 0.4 g/L

$$C(NaOH)_i \times V(NaOH)_i = C(NaOH)_f \times V(NaOH)_f$$

Equation 8

$$320 \times V(NaOH)_i = 0.4 \times 2000$$

Equation 9

$$V(NaOH)_i = 2.5\ L$$

Equation 10

- For 200 ppm of NaClO:

[NaClO]= 0.2g/L

$$C(NaClO)_i \times V(NaClO)_i = C(NaClO)_f \times V(NaClO)_f$$

Equation 11

$$130 \times V(NaClO)_i = 0.2 \times 2000$$

Equation 12

$$V(NaClO)_i = 3.1\ L$$

Equation 13

pH of the solution:

M(NaOH)= 40.00 g/mol

$$[NaOH] = \frac{0.4}{40.00} = 0.01\ M$$

Equation 14

$[OH^-]$= 0.01 M

$$pOH = -\log 0.01 = 2$$

Equation 15

$$pH = 14 - 2 = 12$$

Equation 16

3.4.3 Cleaning with citric acid

The cleaning will be performed using the "production" program and the settings will be chosen according to the membrane cleaning needs. The volume will have to be set for its highest (100 m^3) and the program will have to be stopped when the intended time passes. For the acid cleaning the time should be between 30 and 240 minutes and the backwash interval of time should be between 1 and 3 minutes [27]. This time will be chosen according to the fouling of the membrane. All the other settings will be maintained as the usual values because it will not change anything in the cleaning process.

The harvest tank will be filled with 2000 L of solution in order to keep recirculate the solution during the time needed.

The information from the supplier regarding the cleaning of the membrane with citric acid is that it should be used a solution with a pH equal or higher than 1. After searching for information, the advised concentration is of 0.2 wt% to 0.3 wt% of a citric acid solution [26].

- For a solution of 0.2 wt%:

$\rho(H_2O)$= 997 Kg/m^3

$$m(H_2O) = 997 \times 2 = 1994\ Kg$$

Equation 17

$$m(C_6H_8O_7) = \frac{1994 \times 0.2}{99.8} = 4{,}00\ Kg = 4000\ g$$

Equation 18

M($C_6H_8O_7$)= 192.12 g/mol

$$n(C_6H_8O_7) = \frac{4000}{192.12} = 20.82\ mol$$

Equation 19

$$[C_6H_8O_7] = \frac{21.03}{2000} = 0.011\ M$$

Equation 20

Ka($C_6H_8O_7$)= 8.4x10^{-4} [28]

$$C_6H_8O_7 + H_2O \leftrightarrow C_6H_7O_7{}^{-} + H_3O^{+}$$

Initial (M)	0.011	0	0
Changed (M)	-x	+x	+x
Equilibrium (M)	0.011-x	x	x

$$K_a = \frac{[C_6H_7O_7{}^{-}][H_3O^{+}]}{[C_6H_8O_7]}$$

Equation 21

$$\frac{x^2}{0.011 - x} = 8.4 \times 10^{-4}$$

Equation 22

$$x^2 + 8.4 \times 10^{-4}x - 9.2 \times 10^{-6} = 0$$

Equation 23

$$x = -0.0035\ \vee x = 0.0026$$

Equation 24

[H_3O^+]= 0.0026 M

$$pH = -\log 0.0026 = 2.59$$

Equation 25

- For a solution of 0.3 wt%:

$$m(C_6H_8O_7) = \frac{1994 \times 0.3}{99.7} = 6.00\ Kg = 6000\ g$$

Equation 26

$$n(C_6H_8O_7) = \frac{6000}{192.12} = 31.23\ mol$$

Equation 27

$$[C_6H_8O_7] = \frac{31.23}{2000} = 0.016\ M$$

Equation 28

$$C_6H_8O_7 + H_2O \leftrightarrow C_6H_7O_7{}^- + H_3O^+$$

Initial (M)	0.016	0	0
Changed (M)	-x	+x	+x
Equilibrium (M)	0.016-x	x	x

$$K_a = \frac{[C_6H_7O_7{}^-][H_3O^+]}{[C_6H_8O_7]}$$

Equation 29

$$\frac{x^2}{0.016 - x} = 8.4 \times 10^{-4}$$

Equation 30

$$x^2 + 8.4 \times 10^{-4}x - 1.3 \times 10^{-5} = 0$$

Equation 31

$$x = -0.0040\ \vee x = 0.0032$$

Equation 32

$[H_3O^+]$= 0.0032 M

$$pH = -\log 0.0032 = 2.49$$

Equation 33

- Considering a pH equal to 2.5:

$$[H_3O^+] = 10^{-2.5} = 0.0032M$$

Equation 34

$$C_6H_8O_7 + H_2O \leftrightarrow C_6H_7O_7{}^- + H_3O^+$$

Initial (M)	x	0	0
Changed (M)	-0.0032	+0.0032	+0.0032
Equilibrium (M)	x-0.0032	0.0032	0.0032

$$K_a = \frac{[C_6H_7O_7{}^-][H_3O^+]}{[C_6H_8O_7]}$$

Equation 35

$$\frac{0.0032^2}{x - 0.0032} = 8.4 \times 10^{-4}$$

Equation 36

$$8.4 \times 10^{-4}x = 1.3 \times 10^{-5}$$

Equation 37

$$x = 0.015$$

Equation 38

$[C_6H_8O_7]$= 0.015 M

$$n(C_6H_8O_7) = 0.015 \times 2000 = 30\ mol$$

Equation 39

$$m(C_6H_8O_7) = 30 \times 192.12 = 5.8 \times 10^3\ g = 5.8\ Kg$$

Equation 40

3.4.4 Permeability graphic analysis

To easily analyze the data, the permeability checks were divided in four classes:

- **Class I:** Graphics that the membrane was not completely covered with water so the membrane area cannot be calculated.
    - **a:** The membrane was not completely covered with water and the permeability check did not went as expected (Figure 3.6).

    - **b:** The membrane was not completely covered with water but the permeability check went as expected (Figure 3.7).
- **Class II:** Graphics that are not in line with what is expected.
    - **a:** The permeate flow does not stabilizes throughout the permeability check (Figure 3.8).
    - **b:** The permeate flow oscillates at the begging but eventually stabilizes (Figure 3.9).
- **Class III:** Normal graphics with values of permeability below 90% (Figure 3.10).
- **Class IV:** Normal graphics with values of permeability equal or higher than 90% (Figure 3.10).

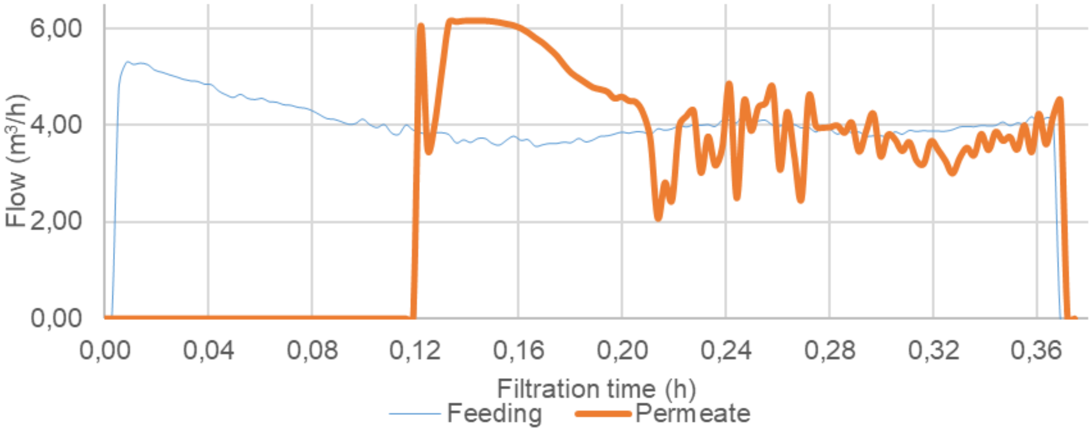

Figure 3.6 – Permeability check class Ia.

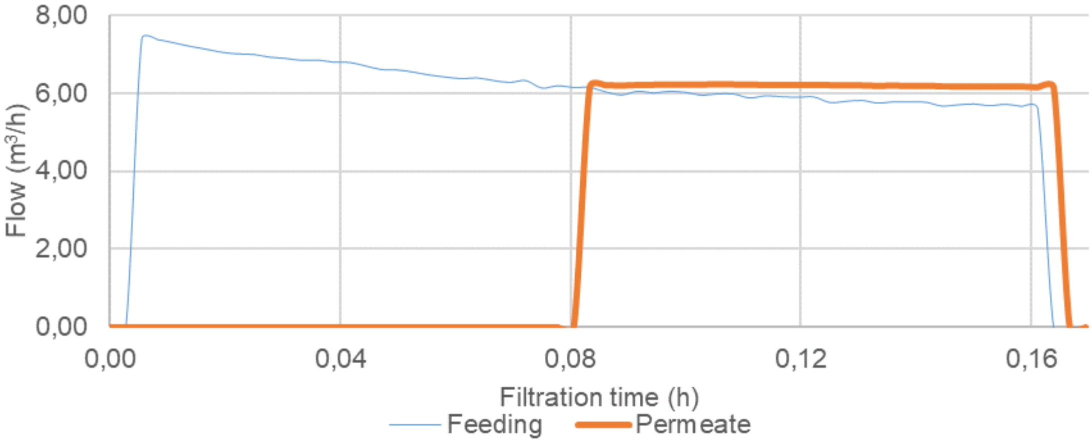

Figure 3.7 – Permeability check class Ib.

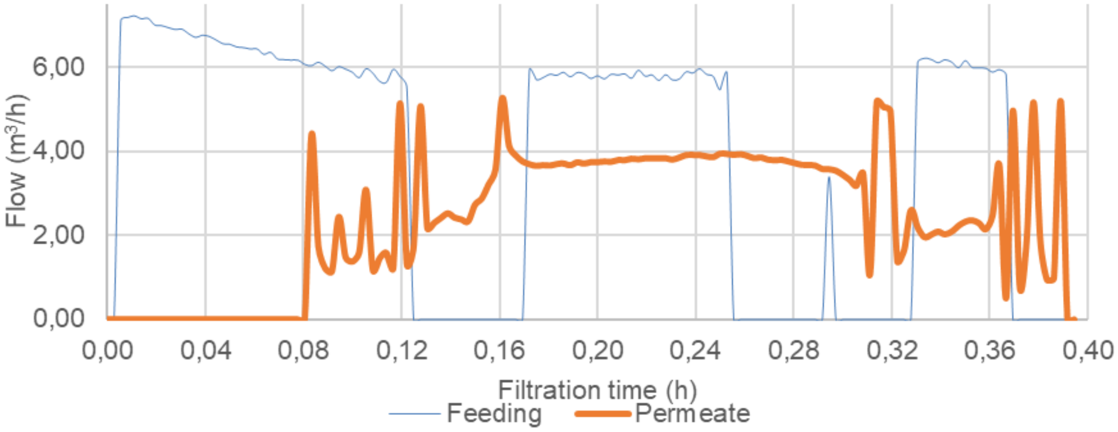

Figure 3.8 – Permeability check class IIa.

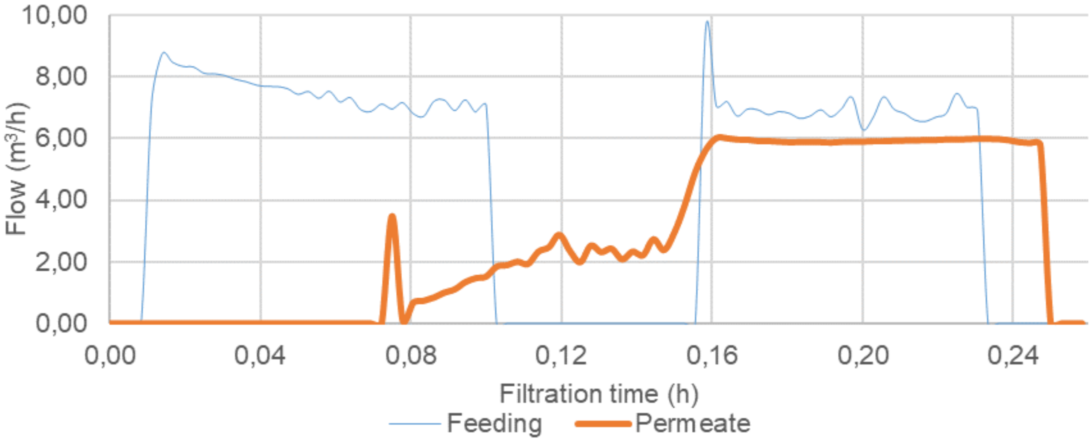

Figure 3.9– Permeability check class IIb.

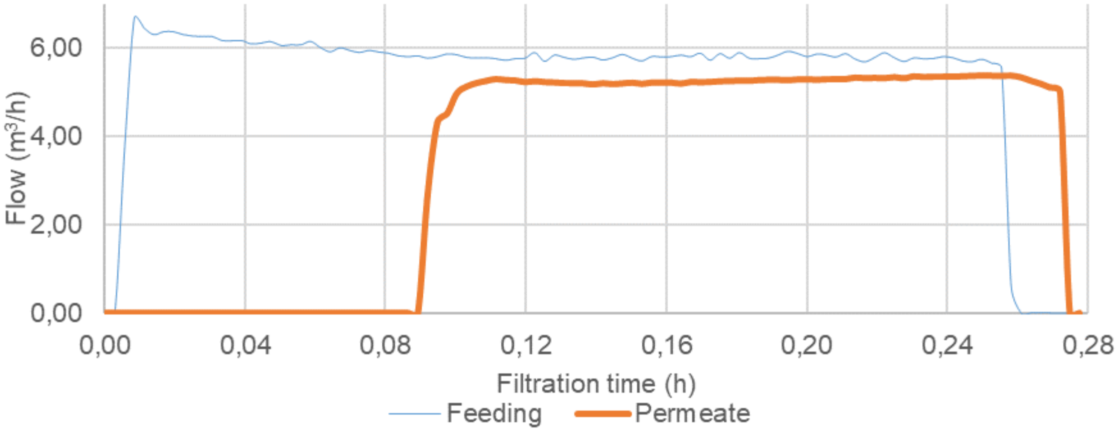

Figure 3.10 – Permeability check class III and IV.

Graphics like Figure 3.6 and Figure 3.7 are considered class I because, as can be seen on the graphic, the permeate flow is higher than the feed flow, so the level on the concentrator tank starts to decrease and part of the membrane starts to be uncovered. For this graphics it is not possible to calculate the membrane permeability because it is impossible to know the area of the membrane that was covered with water and filtrating.

The difference between class Ia and class Ib is that in class Ib the graphic goes accordingly to what it should and in class Ia does not. In Figure 3.6 can be seen that the permeate suffers oscillations, this is most likely due to the air that is sucked into the membranes, by the parts of the membranes that are not covered with water, causing the flow meter to register oscillations on the permeate flow. It can also happen if the system is not completely clean, making the water that is feed to the concentrator tank drag this molecules to the membrane making the permeate flow suffer oscillations. This does not happens in Figure 3.7 and, as can be seen on the graphics, the permeate flow in Figure 3.7 does not have as big as a difference from the feeding flow as it does in Figure 3.6. So most likely the membrane was not as uncovered in Figure 3.7 as it was in Figure 3.6, so the air being sucked to the membrane was not enough to cause oscillations on the permeate flow. And it also might be that the system was clean and the water that was feed to the concentrator tank did not dragged molecules to the membrane.

Graphics like Figure 3.8 and Figure 3.9 are considered class II because, as can be seen on the graphic, the permeate flow suffers oscillations and, unlike the previous graphics, this ones are not due to the membrane being uncovered. So, the most likely reason for this to happen is that the system is not completely clean, so the water that is feed to the concentrator tank drags these molecules to the membrane making the permeate flow suffer oscillations.

The difference between class IIa and IIb is that in class IIb the permeate flow starts by oscillating but eventually stabilizes, so it is possible for the permeability of the membrane to be calculated when the graphic stabilizes. This does not happen in class IIa, the permeate flow keeps oscillating so, for this class, it is impossible to calculate the permeability of the membrane.

Graphics like Figure 3.10 are the type of graphics that should always be obtained when doing a permeability check, they are considerate class III or IV depending on the permeability of the membrane. If the permeability of the membrane is higher than 90% then they are class IV, because it means that the system is well cleaned and ready to filtrate again. If the membrane permeability is lower than 90% it is considered class III, because the system is not well cleaned and needs more cleaning steps in order to restore the membrane permeability to a value equal or higher than 90%.

The difference between graphics that have oscillations in the permeate flow, due to the system not being well cleaned, and graphic that go according to the normal but have a very low membrane permeability, is that when there are oscillations is most likely due to small particles that can pass through the membrane and when there is no oscillations is most likely due to particles that are bigger than the membrane pores, covering part of the membrane and not letting the permeate pass through.

3.4.5 Concentration factor

It also had to be taken into consideration the concentration factor (Equation 41) when filtrating, to compare how much clean it would need two different harvest filtrations of the same algae but with different concentration factors.

$$Concentration\ factor = \frac{Processed\ Volume\ (L)}{Concentrated\ Volume\ (L)}$$

Equation 41

3.5 Harvest filtration

For the harvest filtration the “Production” program is used. The “process volume” in the settings has to be set according to the volume that was harvested and that has to be filtrated. The backwash interval has to be set according to the type of algae that is going to be filtrated and the volume that is going to be filtrated.

A harvest graphic should look like Figure 3.11.

When the part of the graphic that corresponds to the feeding is equal to zero, it means that the feeding stopped. This happens because the level on the concentrator tank reached the LS-06 sensor.

The permeate flow starts when the level on the concentrator tank reaches the LS-05 sensor. After this happens, the points in the graphic that have a permeate flow equal to zero correspond to the backwashes. In this case the backwash was set to happen every 8 minutes as we can see in Figure 3.11.

In the harvest graphics the TMP will continue to go accordingly to the permeate flow (Figure 3.11 and Figure 3.12), so it only important to study the TMP graphics, when the TMP set on the program is changed during the filtration, because some of the filtrations were done while using the centrifuge at the same time, so the TMP set on the setting of the filtration machine would have to be change in order to filtrate at the same rhythm as the centrifuge (Figure 3.13 and Figure 3.14).

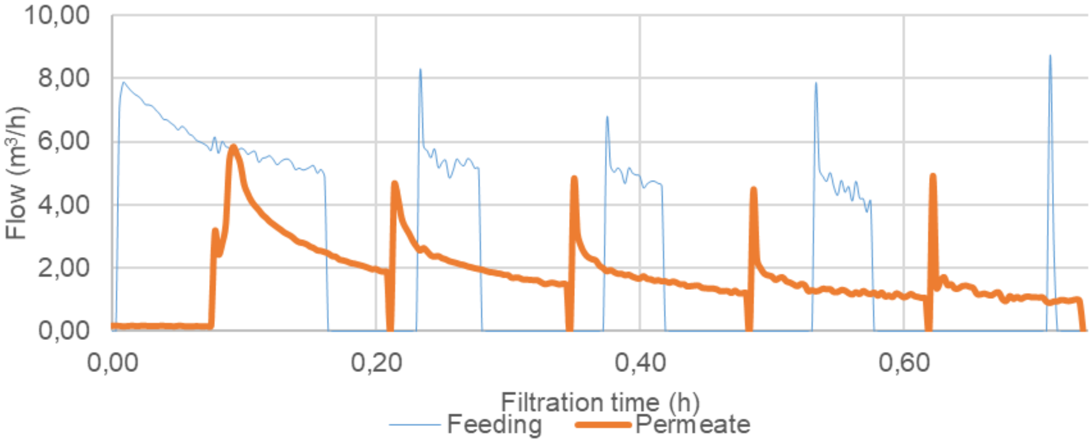

Figure 3.11 – Harvest example.

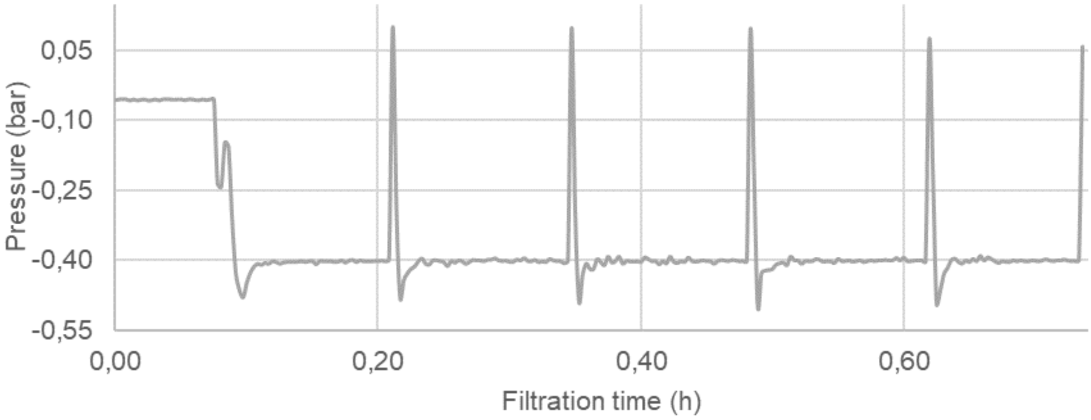

Figure 3.12 – TMP of the harvest filtration of Figure 3.11.

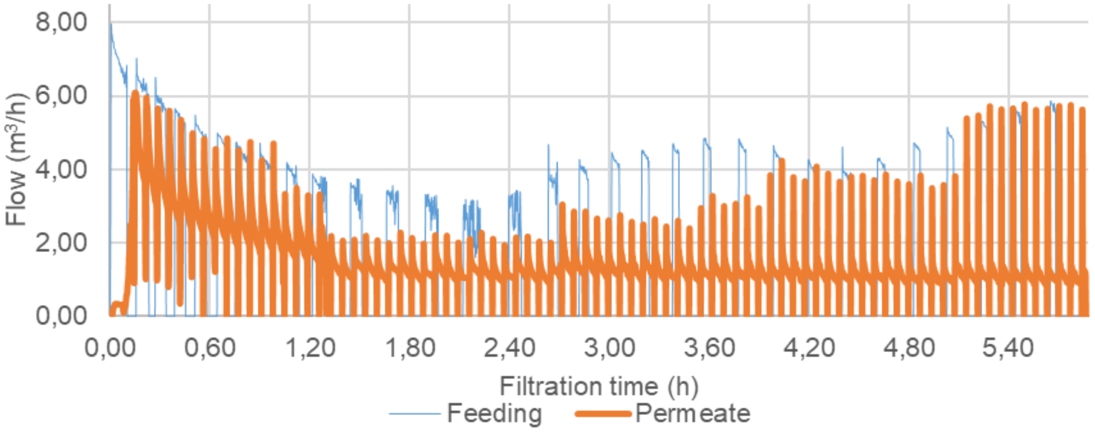

Figure 3.13 – Harvest example.

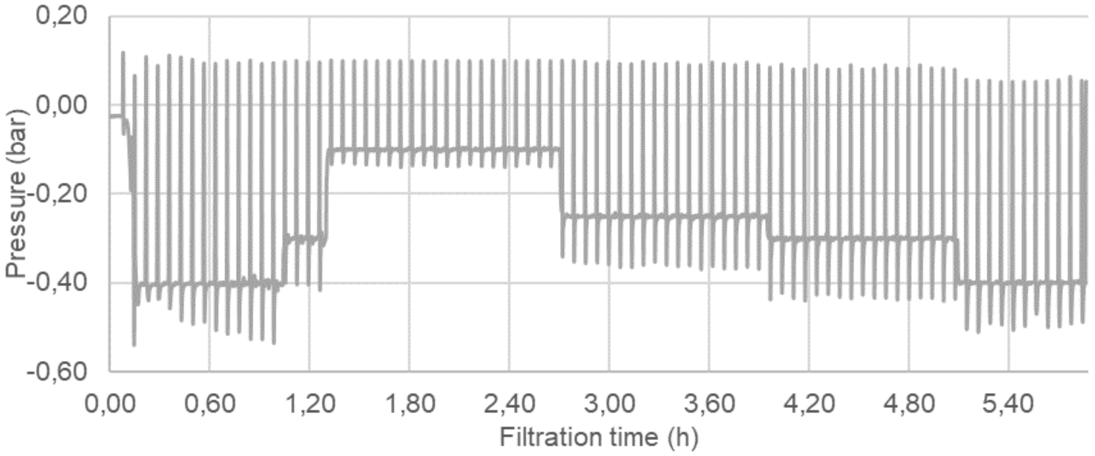

Figure 3.14 – TMP of the harvest filtration of Figure 3.13.

4 Results and discussion

4.1 Cleaning results

The filtration system is experimental and is being tested by A4F. Due to it being an experimental program, it has some flaws that were evaluated and tried to solve during this master thesis.

At first there were four evident problems with the system.

The first problem is that when a “production” program starts, the system automatically empties the concentrator tank. Normally this would not be a problem but if the light goes out during a filtration, when it comes back on, in order to start again a “production” program for the missing volume of harvest culture, the system will automatically throw away all that there is inside the concentrator tank that, in this case, will be the filtrated algae. So, in order to fix this problem, the supplier should change the “production” program and add a question in the begging of the “production” program, asking if the system should throw away what was inside the concentrator tank or not. For the time being the existing solution is a bypass on the drain pipe for the concentrate to go back to the feeding tank, but it is not ideal because it consumes more time.

The second problem is that there is no bottom purge on the permeate tank. The only way to remove the content of the tank is through a pipe on its side (Figure 3.1), making it impossible to completely remove the content of it. For example, if a cleaning with acid is performed, then the permeate tank will contain acid which is not good when the idea is to do a filtration next and use the permeate to produce culture medium. In order to solve this, the supplier should add a bottom purge for when the content of the permeate tank needed to be completely removed. Right now the solution is to always dilute what is inside the tank, for example after a cleaning with acid, all that is possible to remove from the permeate tank is removed and water is added to the tank in order to dilute what was impossible to remove, after that the water is also removed until the level on the tank reaches sensor LS-01.

The third problem is that when the level on the permeate tank goes below the sensor LS-01,for example if is needed for the content on the permeate tank to be removed using the manual mode, an error on the system shows up and it will not let any operation on the machine continue to be performed. This would not be a problem if there was a way to fill the tank again until it reached the sensor, but there is no way to fill it. The solution for this was for a water pipe to be directly connected to the permeate tank, in order to fill it when the level goes bellow the sensor. For the time being the solution that there is, is to have a hose inside the permeate tank that has to be manually turned on when the level is under sensor LS-01. This is not an ideal solution because it cannot be done automatically and implicates that the permeate tank always needs to have a small opening on the cover for the hose to be inside making it easier to get contaminated.

The fourth problem is that when the concentrator tank is filled until the sensor LS-06, if the blower is not turned on, water starts filling the pipe that takes air from the blower to the membrane, making it possible for water to get into the blower and damage it. There is a valve that can be closed when the blower is not being used, this way if the concentrator tank was to be filled until the LS-06 sensor, water will not go into the blower, but if the blower is needed it should be turned on when the level on the concentrator tank reaches the sensor LS-05. This will still be a problem if the light goes out and the concentrator tank is filled until the sensor LS-06. In order to solve this, the pipe that connects the blower to the membrane should be higher than the concentrator tank, that way, even if water would go into the pipe, it would never reach the blower. The circle in Figure 4.1 is where the blower is connected to the concentrator tank and the green lines is how it should be connected in order for the water not to go into the blower.

Figure 4.1 – Blower connection to the concentrator tank.

The results on Table 4.1 were done before this master thesis and I was not present in the cleaning after the filtration of the 5th of March, so the cleaning steps are not as detailed as after I started working in the optimization of the system, but the analyze of the results after the different steps of cleaning was useful to comprehend the data and plan the new steps for the following cleanings.

Table 4.1 – Cleaning results.

Processing Date	Microalgae	Concentration Factor	CIP Cycles	Permeability Date	Average Permeate Flow (m^3/h)	Average TMP (bar)	Permeability (L/(m^2 h bar))	Recovery	Class	Cleaning Steps
02.09.2019	*Dunaliella*	13,00	2	06.09.2019	3,88	-0,33	219	37%	IIb	F→4CS→20CIP→20CIP→PC→4CS→PC→PC
				10.09.2019	1,95	-0,21	215	37%	IIb	
				10.09.2019a	4,23	-0,39	197	34%	III	
12.09.2019	*Nannochloropsis*	5,03	2	13.09.2019	4,85	-0,39	230	39%	III	F→1CS→20CIP→20CIP→PC
27.09.2019	*Nannochloropsis*	5,35	2	30.09.2019	1,99	-0,18	225	38%	IIb	F→20CIP→20CIP→3CS→PC→PC
				30.09.2019a	5,15	-0,40	-	-	Ib	
25.10.2019	*Dunaliella*	8,29	1	29.10.2019	1,44	-0,20	146	25%	IIb	F→20CIP→4CS→PC→1CS→PC
				30.10.2019	4,29	-0,41	198	34%	III	
11.12.2019	*Nannochloropsis*	14,28	3	13.12.2019	3,08	-0,36	176	30%	IIb	F→20CIP→20CIP→20CIP→2CS→PC→3CS→PC →3CS→PC→11CS→PC
				16.12.2019	3,81	-0,35	208	35%	IIb	
				19.12.2019	5,32	-0,40	247	42%	III	
				30.12.2019	6,23	-0,36	322	55%	III	
02.01.2020	*Nannochloropsis*	6,52	3	06.01.2020	5,68	-0,30	353	60%	IIb	F→20CIP→20CIP→20CIP→4CS→PC→PC→4CS →PC
				06.01.2020a	6,18	-0,37	-	-	Ib	
07.01.2020	*Nannochloropsis*	17,56	3	10.01.2020	4,84	-0,33	-	-	Ia	F→20CIP→1CS→20CIP→20CIP→1CS→PC
16.01.2020	*Odontella*	3,61	3	17.01.2020	5,34	-0,32	-	-	Ia	F→20CIP→20CIP→20CIP→1CS→PC→4CS→PC →PC→1CS→PC
				21.01.2020	5,60	-0,27	402	69%	IIb	
				21.01.2020a	5,82	-0,38	-	-	Ib	
				22.01.2020	6,20	-0,37	311	53%	III	
23.01.2020	*Nannochloropsis*	4,23	3	24.01.2020	4,10	-0,29	274	47%	IIb	F→20CIP→20CIP→20CIP→1CS→PC→PC
				24.01.2020a	5,90	-0,40	275	47%	III	
28.01.2020	*Nannochloropsis*	6,83	3	30.01.2020	3,85	-0,23	319	54%	IIb	F→20CIP→20CIP→20CIP→2CS→PC→PC→1CS →PC→7CS→PC→1CS→2BC10
				30.01.2020a	5,98	-0,40	-	-	Ib	
				31.01.2020	5,60	-0,39	-	-	Ib	
				07.02.2020	4,85	-0,34	267	46%	IIb	
05.03.2020	Supernatant *Odontella*	2,68	1	06.03.2020	5,09	-0,34	-	-	Ia	F→20CIP→1CS→PC→PC→5CS→PC
				06.03.2020a	5,34	-0,40	-	-	Ib	
				11.03.2020	5,90	-0,38	-	-	Ib	

After analyzing the data from Table 4.1 the main steps performed in the cleaning process were the CIP, always with a soaking time of 20 minutes and leaving the system filled with water and 200 ppm of sodium hypochlorite. There were three other steps very common but never described when they were used or how many times they were used in the previous data, this steps were washing with a hose (WH), "Production" program with recurrent backwashes (BC) and passing water through the pipes to remove the algae that got stuck in there, that included closing and opening the valves to clean them too.

In Table 4.2 can be seen a more detailed cleaning, when I started working with system. In between the cleaning steps, the water valve would be opened, with the valve VA-04 opened in order to cleaning the bottom of the concentrator tank. Water would also be passed through the pipes to clean them after every filtration. This were steps that should always be done so they were not described in the cleaning steps.

Table 4.2 – Cleaning results.

Processing Date	Microalgae	Concentration Factor	CIP Cycles	Permeability Date	Average Permeate Flow (m^3/h)	Average TMP (bar)	Permeability (L/(m^2 h bar))	Recovery	Class	Cleaning Steps
13.02.2020	Supernatant *Nannochloropsis*	6,55	1	14.02.2020	5,95	-0,40	279	48%	III	F→WH→20CIP→1CS→2BC10→PC
11.03.2020	*Nannochloropsis*	9,25	4	12.03.2020	5,19	-0,34	-	-	Ia	F→WH→20CIP→WH→CF→1CSS→WH→BW→BW→BW→WH→20CIP→2BC10→BW→BW→WH→BW→20CIP→PC→PC→1CSS→PC→PC→BW→BW→BW→BW→BW→PC→3CAS→BW→PC→20CIP→2BC10→73CSS→PC→PC
				12.03.2020a	5,00	-0,40	-	-	Ib	
				13.03.2020	4,49	-0,39	-	-	Ia	
				13.03.2020a	4,28	-0,36	-	-	Ia	
				13.03.2020b	4,34	-0,34	-	-	Ia	
				16.03.2020	4,28	-0,34	-	-	Ia	
				28.05.2020	5,61	-0,23	-	-	Ia	
				28.05.2020a	5,22	-0,37	-	-	Ib	

A new step was added, backwashing with the pump at 50 Hz (BW), when this step was performed a lot of dirty was seen coming out of the membrane, so at first it seemed like a good cleaning step.

The cleaning performed after the filtration on the 11th of March took several days and after all of these days, even though the water coming out of the concentrator tank was coming clean, the permeability checks were still not good, so when we reached the top of the membrane and touched in between the bundles to see if it was really clean (what came out can be seen on Figure 4.2), we realized it was not and the cleaning steps were not being thorough enough. This got us to the conclusion that maybe all the steps done during the cleanings in the past were never enough to get the membrane actually clean.

On the other hand, cleaning the system after a filtration of supernatant that came from the centrifuge, was much easier to get a class III permeability check.

So after analyzing all this new data, it was concluded that the filtration of the harvest was not time efficient, the membrane would get very dirty and it would take several days to clean it, so it was decided that it would be more efficient if the filtration system was used after the culture had been passed through the centrifuge, using then the filtration system to filtrate the supernatant and reuse it to make more culture medium for the algae. This would mean less cleaning steps, less time consuming cleaning and more efficient ones, as we can see from the cleaning performed after the filtration on the 13th of February.

Figure 4.2 – Dirt that came out of the membrane after several days of cleaning.

In Table 4.3 are the results from the fitrations of the supernatant coming from the centrifuge.

Table 4.3 – Cleaning results.

Processing Date	Microalgae	Concentration Factor	CIP Cycles	Permeability Date	Average Permeate Flow (m^3/h)	Average TMP (bar)	Permeability ($L/(m^2\ h\ bar)$)	Recovery	Class	Cleaning Steps
29.05.2020	Supernatant *Nannochloropsis*	3,66	3	29.05.2020	3,99	-0,36	-	-	Ia	F→BW→WH→20CIP→PC→WH→20CIP→BW→WH→BW→BW→3CS→WH→20CIP→BW→WH→BW→BW→WH
02.06.2020	Supernatant *Nannochloropsis*	5,73	2	02.06.2020	3,43	-0,30	-	-	Ia	F→WH→20CIP→WH→BW→BW→WH→20CIP→PC→MB
03.06.2020	Supernatant *Nannochloropsis*	17,71	1	03.06.2020	3,69	-0,40	-	-	Ia	F→BW→MB→PC
04.06.2020	Supernatant *Nannochloropsis*	21,07	4	05.06.2020	4,87	-0,40	-	-	Ib	F→BW→WH→1CS→WH→20CIP→WH→BW→PC→PC→BW→20CIP→MB→BW→PC→4CS→MB→BW→PC→60CIP→MB→PC→40CIP→MB→PC→PC→BW→BW→PC
				05.06.2020a	4,59	-0,40	-	-	Ia	
				05.06.2020b	4,72	-0,40	-	-	Ia	
				09.06.2020	4,86	-0,35	-	-	Ia	
				09.06.2020a	5,80	-0,39	-	-	Ib	
				09.06.2020b	6,20	-0,34	-	-	Ib	
				09.06.2020c	4,89	-0,35	-	-	Ia	
				09.06.2020d	3,84	-0,27	-	-	Ia	

From observing Table 4.2 and Table 4.3 we were able to see that every time that a backwash with the pump at 50 Hz (BW) was performed right before a permeability check, the permeate pump would start working when the concentrator tank level reached the LS-05 sensor but there was almost no flow of permeate passing through for a long time, the permeate flow would increase very slowly which is not supost to happen (Figure 4.3). So even though, when performing a backwash with the pump at 50 Hz we would see a lot of dirt coming out of the membrane, this is not a good cleaning step. To replace this step it would be better to always do the manual backwash cleaning (MB) with the concentrator tank full of water, since the backwash with the pump at 50 Hz would be without any water on the tank.

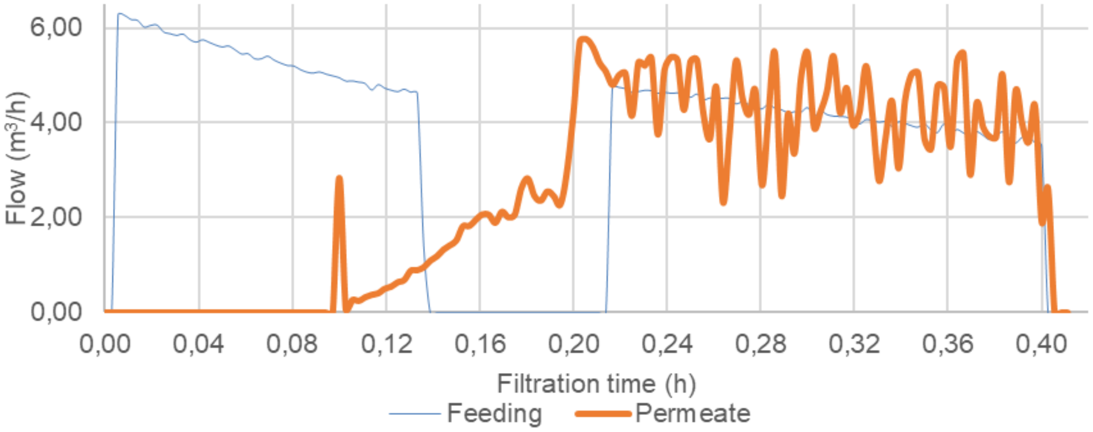

Figure 4.3 – Permeability check 02.06.2020.

In Table 4.4 are the results from the fitrations of the supernatant coming from the centrifuge after the step of backwashing with the pump at 50 Hz (BW) was removed.

Table 4.4 – Cleaning results.

Processing Date	Microalgae	Concentration Factor	CIP Cycles	Permeability Date	Average Permeate Flow (m^3/h)	Average TMP (bar)	Permeability ($L/(m^2\ h\ bar)$)	Recovery	Class	Cleaning Steps
18.06.2020	Supernatant *Nannochloropsis*	19,21	2	22.06.2020	4,10	-0,39	-	-	Ia	F→WH→FT→MB→4CSS→WH→60CIP→1BC15→MB→PC→60CIP→MB→1BC15→PC
				22.06.2020a	3,38	-0,38	-	-	Ia	
01.07.2020	Supernatant *Nannochloropsis*	17,42	1	01.07.2020	1,90	-0,40	-	-	IIa	F→FT→60CIP→PC
02.07.2020	Supernatant *Nannochloropsis*	19,71	2	03.07.2020	3,10	-0,38	-	-	IIa	F→WH→1CS→FT→60CIP→FT→PC→MB→PC→0,0625CS→FT→MB→PC→60CIP→FT→MB→PC→7CS→FT→MB→PC→240CAC3→FT→MB→PC→PC
				03.07.2020a	3,82	-0,39	181	31%	III	
				03.07.2020b	4,26	-0,40	201	34%	III	
				03.07.2020c	4,35	-0,40	205	35%	III	
				10.07.2020	6,08	-0,39	-	-	Ib	
				10.07.2020a	5,58	-0,39	-	-	Ib	
				10.07.2020b	4,03	-0,20	370	63%	III	

In Table 4.3 and Table 4.4 the CIP time was increased in order to see if the permeability would increase, but it did not so a cleaning step, the cleaning with citric acid (CAC), was added and it can be seen that the permeability increased from 205 $L/(m^2\ h\ bar)$ to 370 $L/(m^2\ h\ bar)$.

After the filtration the concentrator tank was washed with a hose (WH) to remove most of the dirt and then a CIP was performed, but cleaning just with a hose would not remove the dirt from the membrane and it would not be enough so after cleaning with a hose, the tank started to be filled with water and the blower turned on (FT) in order for the water to get to every place of the tank and remove as much dirt as possible.

Another problem was that the feed pump P-03 had a leak and this would cause the permeate flow to be higher than the feed flow, because the pump was not able to have enough pressure to keep the flow as high or higher than the permeate flow so the level in the concentrator tank would go below sensor LS-05 making part of the membrane to be uncovered. This way it was impossible to know what was the area of the membrane that was filtrating, making it impossible to know the membrane permeability. The way that was used to go around this issue was to change the usual TMP of -0.4 bar to -0.2 bar, this way the permeate is not sucked as fast as it was before and it is possible to obtain the permeability. One thing that was also taken into account was that the volume on the feeding tank should always be higher than 2000L in order to help the feed pump to have some more pressure from gravity. This started to be done in the permeability check of 10.07.2020b.

In

Table 4.5 are the results from the fitrations of the supernatant coming from the centrifuge after the new steps described above were added.

Table 4.5 – Cleaning results.

Processing Date	Microalgae	Concentration Factor	CIP Cycles	Permeability Date	Average Permeate Flow (m^3/h)	Average TMP (bar)	Permeability ($L/(m^2$ h bar))	Recovery	Class	Cleaning Steps
13.07.2020	Supernatant *Nannochloropsis*	3,19	1	13.07.2020	3,31	-0,20	308	53%	III	F→FT→20CIP→FT→MB→PC→15CS→MB→PC→240CSC3→FT→MB→PC→1CS→FT→MB→PC
				28.07.2020	5,19	-0,20	494	84%	III	
				28.07.2020a	4,34	-0,20	-	-	Ib	
				29.07.2020	5,84	-0,21	530	91%	IV	
29.07.2020	Supernatant *Nannochloropsis*	5,71	0	29.07.2020a	3,71	-0,20	362	62%	III	F→FT→FT→FT→MB→PC→1CS→MB→FT→MB→PC→PC→13CS→MB→FT→MB→PC
				30.07.2020	4,90	-0,20	-	-	Ib	
				30.07.2020a	5,75	-0,21	505	86%	III	
				12.08.2020	6,04	-0,22	519	88%	III	
12.08.2020	Supernatant *Nannochloropsis*	4,69	0	12.08.2020a	4,60	-0,31	281	48%	III	F→WH→FT→MB→FT→MB→FT→MB→PC→1CS→MB→FT→MB→FT→MB→PC
				13.08.2020	5,67	-0,26	403	69%	III	
13.08.2020	Supernatant *Odontella*	1,70	1	13.08.2020a	5,71	-0,29	373	64%	III	F→WH→FT→MB→FT→MB→PC→30CSC2→FT→MB→FT→MB→FT→MB→PC→4CS→MB→FT→MB→FT→MB→PC
				13.08.2020b	5,79	-0,23	463	79%	III	
				17.08.2020	6,10	-0,24	486	83%	IV	
17.08.2020	Supernatant *Nannochloropsis*	4,25	0	17.08.2020b	5,84	-0,37	341	58%	III	F→WH→FT→MB→FT→MB→FT→MB→PC→2CS→MB→FT→MB→FT→MB→PC
				19.08.2020	6,02	-0,25	459	78%	III	
19.08.2021	Supernatant *Nannochloropsis*	6,32	1	19.08.2020a	4,30	-0,40	200	34%	III	F→WH→FT→MB→FT→MB→FT→MB→PC→1CS→MB→FT→MB→FT→MB→60CSC2→FT→MB→FT→MB→FT→MB→PC
				20.08.2020	6,04	-0,31	369	63%	III	
				21.08.2020	6,06	-0,29	392	67%	III	
21.08.2020	Supernatant *Nannochloropsis*	3,06	0	21.08.2020a	4,41	-0,31	266	45%	III	F→WH→FT→MB→FT→MB→FT→MB→PC→3CS→MB→FT→MB→FT→MB→PC
				24.08.2020	6,05	-0,29	388	66%	III	
24.08.2020	Supernatant *Nannochloropsis*	4,95	1	24.08.2020a	5,40	-0,41	245	42%	III	F→WH→FT→MB→FT→MB→FT→MB→PC→1CS→MB→FT→MB→120CAC2→FT→MB→FT→MB→FT→MB→9CS→MB→FT→MB→FT→MB→PC→4CS→MB→FT→MB→FT→MB→PC
				03.09.2020	6,14	-0,20	554	95%	IV	
				07.09.2020	6,15	-0,19	586	100%	IV	

Even though measures were taken to prevent the membrane to be uncovered during the permeability checks, on the second one performed on the 28th of July and on the first one performed on the 30th of July this still happened. So later, on the 18th of August, the pump was replaced and the feed flow increased exponentially, the difference can be seen in Figure 4.4 (a permeability check done before the pump was replaced) and Figure 4.5 (a permeability check done after the pump was replaced).

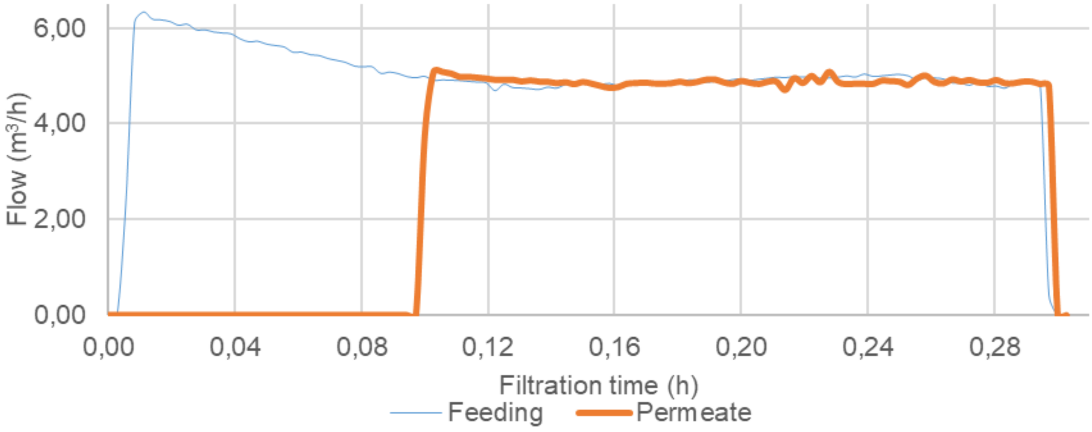

Figure 4.4 – Permeability check 05.06.2020.

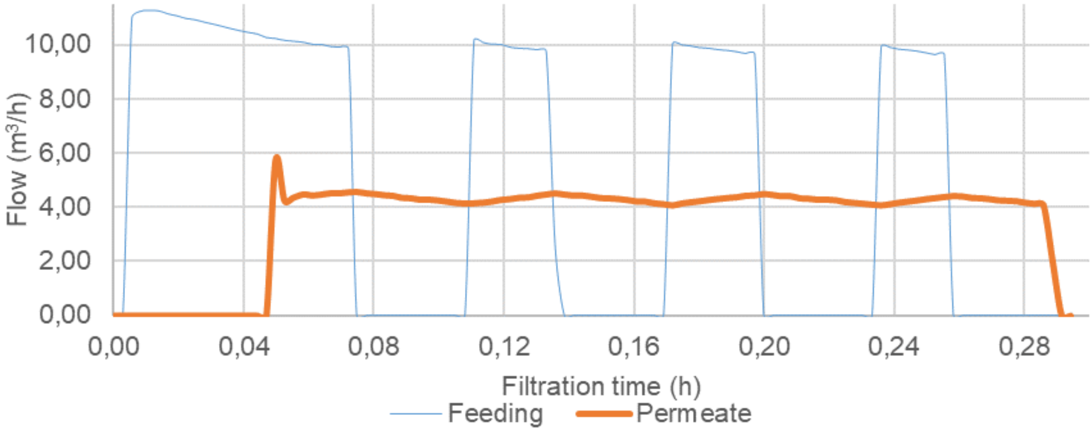

Figure 4.5 – Permeability check 19.08.2020a.

Another problem with the system is the way the CIP program works. The CIP program fills the concentrator tank with water and sodium hydroxide and turns on the blower, so the cleaning solution does not pass through the membrane and into the permeate tank and it also does not perform backwashes, being those very important steps in the cleaning of the membrane. In order to solve this, the supplier should change the CIP program in order for the cleaning solution to pass through all the system and to perform backwashes. For the time being the solution being used to fill the concentrator tank with water and the cleaning solution, then perform a "production" program, setting the maximum volume the system allows (100 m^3) and the backwash interval of time and them leaving the system cleaning during the needed time, when the time passes, the cleaning process has to be stopped manually. This is also a problem because there is no option to choose the time filtrating instead of the filtration volume. The way to solve this would be for the supplier to fix the CIP program or to add the option of choosing to set the filtration volume or the time filtrating.

So the CIP program was replaced by a "Production" program with a solution of 0.04% of sodium hydroxide with 200 ppm of sodium hypochlorite and, as it can be seen in

Table 4.5, the permeability increased to 91% (permeability check 29.07.2020).

In order to see if the membrane is already being efficiently cleaning, due to the water coming clean after the cleaning after the filtration of 11th of March and then the membranes being dirty, the membranes were removed to see their condition. As can be seen in Figure 4.6 the membrane was pretty much clean, the only part that could be seen that was not entirely clean was the top and bottom of the membrane, where the membrane is glued to the support (Figure 4.7), but since the space in between the bundles is very tight we were not able to clearly see the dirt, it was only possible to see that that part was darker than the rest of the membrane. This takes us to another problem with the membrane, since this part of the membrane is so tight is nearly impossible to get it well cleaned, so it would be much more efficient if the part of the membrane that glues to the support was inside of the support and not exposed to the culture that is put inside the concentrator tank, this way the cleaning of the membrane would be much more efficient.

Figure 4.6 – Membrane removal.

Figure 4.7 – Top and bottom of the membrane that is glued to the support.

Another thing that was noticed was that some of the bundles were torn up, about eight bundles taking into account both membranes. The bundle that was in worst shape can be seen in Figure 4.8, the other ones were not as bad and some examples of those can be seen in Figure 4.9.

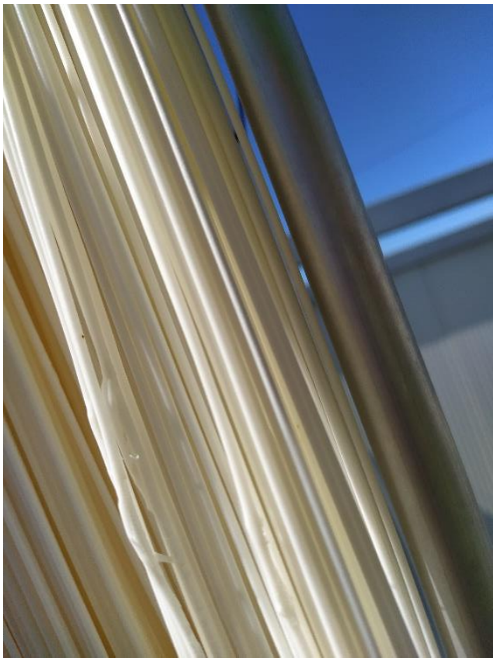

Figure 4.8 – Bundle torn up.

Figure 4.9 – Bundles torn up.

The number of membrane fibers that are torn up are about 0.14% of all the bundles, this explains why the laboratory results show some microalgae cells in the permeate. The first time there is information that cells of microalgae were present in the permeate is the 11th of September of 2019, so this has been happening for some time already and that explains why it was so hard to clean the membrane after a filtration of *Nannochloropsis*.

Nannochloropsis is the smallest microalgae that has been filtrated, has a spherical or oval shape with dimensions of 2-4 x 3-5 µm [29] *Odontella* has a diameter of 10-95 µm [30] and Dunaliella has dimensions of 5-25 x 3-13 µm [31], so it is the easiest cell to pass through the holes that the membrane has. Only cells of *Nannochloropsis* were seen in the permeate so it is safe to assume that only *Nannochloropsis* is able to pass through the membrane.

In order to see the dead spots on the system when cleaning, the pipes were removed to see if there was dirt or biofilms and the only places that in fact there were some biofilms were in the pipes that are connected to the bottom of the concentrator tank, meaning water pipe, concentrator drain and CIP drain, this places can be easily identified in Figure 4.10.

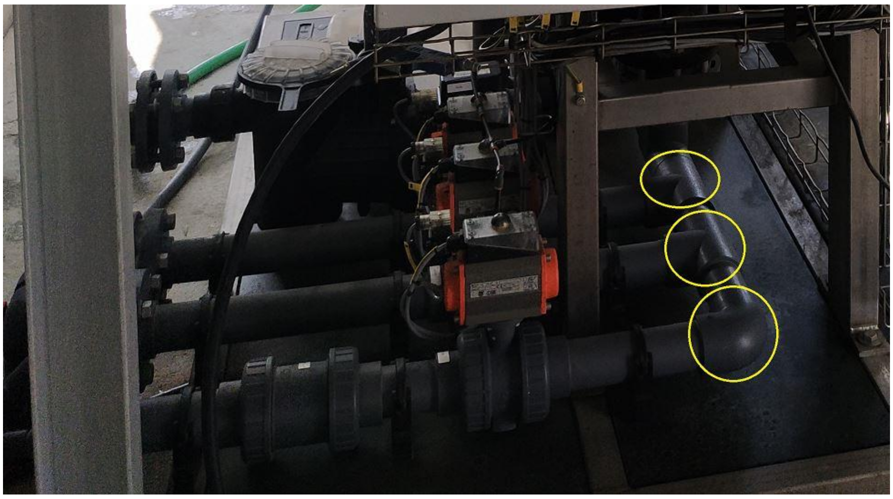

Figure 4.10 – Dead spots of the system.

In Figure 4.11 can be seen the dirt that was inside the pipes and observed that there is almost no dirt in all of the system. In order to try and remove this biofilms, since it is impossible to reach the place where there are biofilms, water pressure was used, but it was not possible to remove them as can be seen in Figure 4.12, after washing the pipes with water pressure.

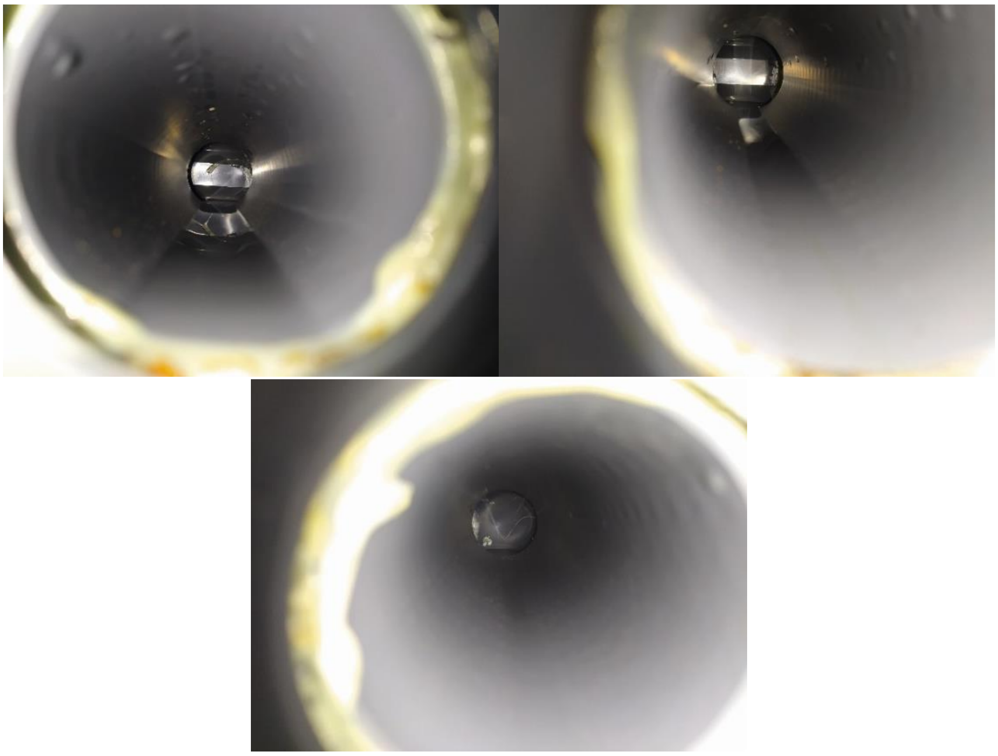

Figure 4.11 – Dirt on the dead spots of the system.

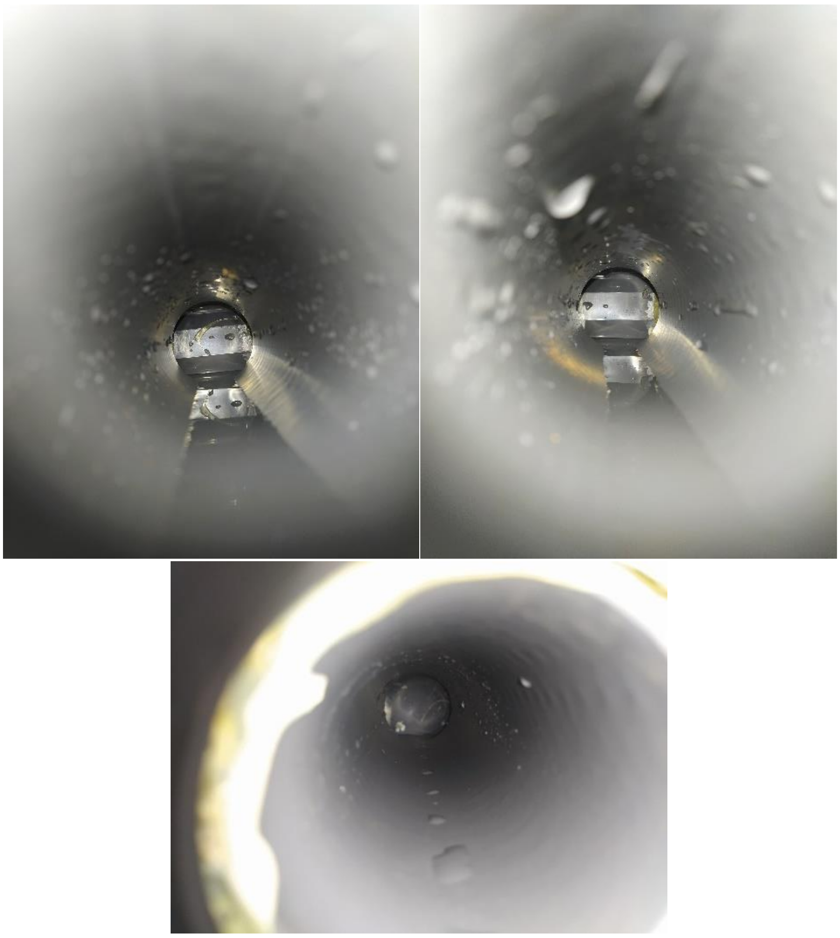

Figure 4.12 – Dead spots after being cleaned with water pressure.

4.2 Harvest optimization

When performing a harvest filtration (“Production” program of the system) the final volume in the concentrator tank is always the same, 466L, so no matter how much volume of harvest we need to filtrate, the final volume will always be the same, what changes is how concentrated that volume at the end is. If we have 10 m^3 to filtrate or 1 m^3 to filtrate, the final concentration of the 10 m^3 filtration will be much higher. So it is important to see until which concentration is viable to use the filtration system.

Another point that has to be taken into consideration is the time in between backwashes, if we have a large volume to process then that time should be smaller than if we have a small volume. It also should be taken into account if the time is not too small that will make the filtration take more time than it should. This time was set by the company to always be 8 minutes so before my master thesis the time used was always 8 minutes.

It should also be considered the duration of the backwash since longer backwashes means more permeate that will have to be filtrated again but, on the other side, it can be a really small backwash that will not be enough for the permeate flow to increase. This time was set by the company to always be 10 seconds so before my master thesis the time used was always 10 seconds.

Since *Nannochloropsis* was at the time the most produced algae in A4F, is the one there is more information about its filtration, but since there were few filtrations of *Nannochloropsis* directly without being the supernatant coming from the centrifuge, there is much more information about the filtration of *Nannochloropsis* supernatant.

In Table 4.6 can be found all the results from the four filtrations done with *Nannochloropsis*.

Table 4.6 – Nannochloropsis harvest filtration results.

Processing Date	Microalgae	Concentration Factor	Average Permeate Flow (m^3/h)	Time	Backwash (min)	Backwash Duration (s)	Average TMP (bar)	Permeability Check Done Before			Observations
								Permeability Date	Recovery	Class	
02.01.2020	*Nannochloropsis*	6,52	0,76	04h:26	8	10	-0,40	30.12.2019	55%	III	PC→3CS→F
23.01.2020	*Nannochloropsis*	4,23	1,50	01h:17	8	10	-0,35	22.01.2020	53%	III	PC→1CS→F
28.01.2020	*Nannochloropsis*	6,80	0,85	03h:48	8	10	-0,38	24.01.2020a	47%	III	PC→4CS→F
11.03.2020	*Nannochloropsis*	9,25	1,44	03h:16	4	10	-0,38	11.03.2020	-	Ib	PC→F

From analyzing Table 4.6 we can see that if the time in between backwashes is diminishes from 8 to 4 minutes then were able to filtrate a higher concentration in lesser time than a smaller concentration, as it is the case of the filtration of 11th of March in which the concentration factor was 9.25 and it took lesser time than the filtration on 28th of January in which the concentrator factor was 6.80.

Since right after the first filtration of *Nannochloropsis* I performed, it was decided that the microalgae would always pass through the centrifuge first, it was not possible to evaluate the duration of the backwash and see if the time was lesser or higher if it would have make a difference or not.

As for the highest concentration factor viable for the system, the filtration with the highest concentration factor is in the 11th of March and the factor is 9.25, from what can be seen in Figure 4.13 the permeate flow is higher right after a backwash and diminishes right after it, as it is normal because the system works by starting the pump and reaching the maximum of 50 Hz but since at that rate the TMP will be higher than -0.4 bar that is what is set to be the highest TMP allowed, the system will than diminish its velocity in order to maintain the TMP below -0.4 bar (Figure 4.14), but throughout the filtration the permeate flow does not changes much and the time that it took to filtrate (three hours and sixteen minutes) was not much. So, it is possible to conclude that until this concentrator factor is viable to filtrate, but since no more filtration were done directly with *Nannochloropsis* and with a higher concentrator factor, it is not possible to conclude until which concentrator factor is viable to filtrate.

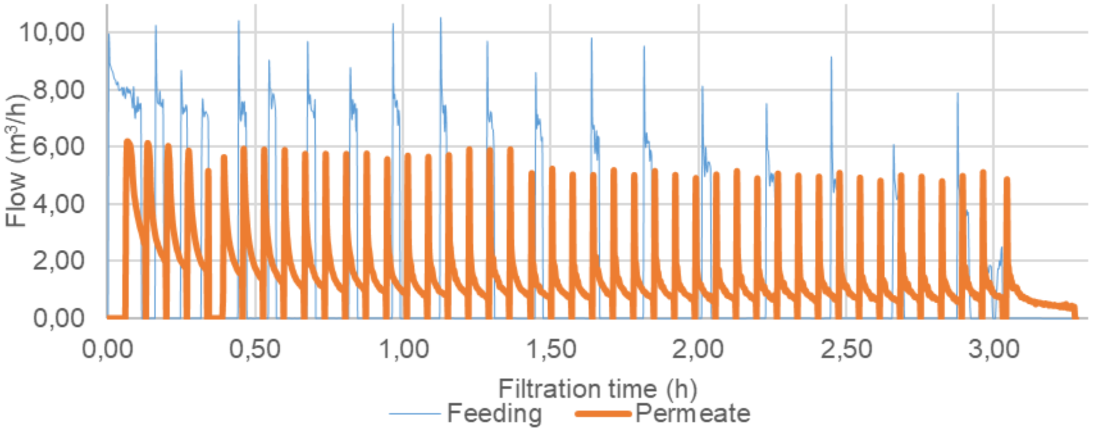

Figure 4.13 – Nannochloropsis harvest filtration 11.03.2020.

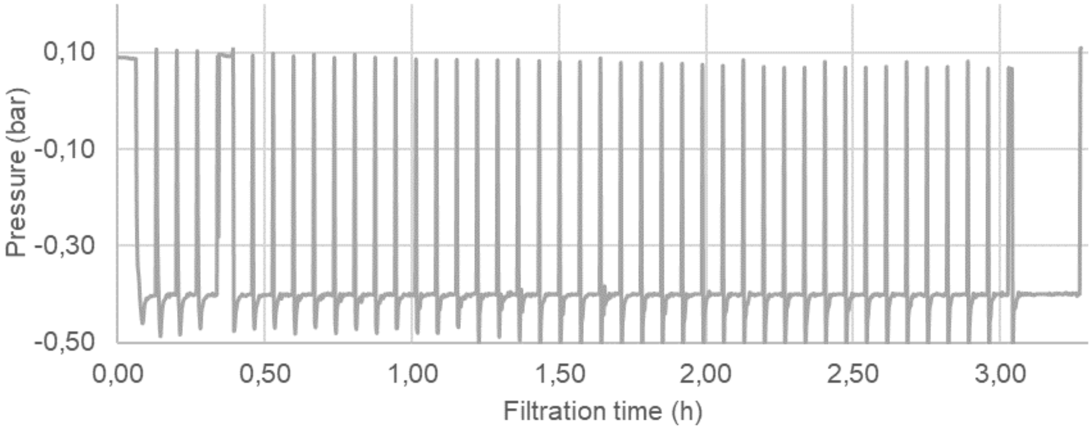

Figure 4.14 – TMP in function of the time for the permeability check on Figure 4.13.

In Table 4.7 can be seen all the results from the two filtrations performed with *Odontella*.

Table 4.7 – Odontella harvest filtration results.

Processing Date	Microalgae	Concentration Factor	Average permeate flow (m^3/h)	Time	Backwash (min)	Backwash Duration (s)	Average TMP (bar)	Permeability Check Done Before			Observations
								Permeability Date	Recovery	Class	
10.12.2019	*Odontella*	6,97	1,36	02:05	8	10	-0,37	-	-	-	20CIP→6CS→F
16.01.2020	*Odontella*	3,61	1,90	00:44	8	10	-0,39	10.01.2020	-	Ia	PC→6CS→F

Since *Odontella* was only directly filtrated before my master thesis started the data is only with the backwash interval of time and backwash duration predefined by the company, so it is not possible to know if diminishing it or increasing it would make it more or less efficient.

As for the concentration factor the highest factor tried was 6.97 and, as can be seen in Figure 4.15, the permeate flow does not changes much from the beginning to the end of the filtration and the time that it took to filtrate (two hours and five minutes) was not much. So it is possible to conclude that until this concentrator factor is viable to filtrate, but since no more filtration were done directly with *Odontella* and with a higher concentrator factor, it is not possible to conclude until which concentrator factor is viable to filtrate.

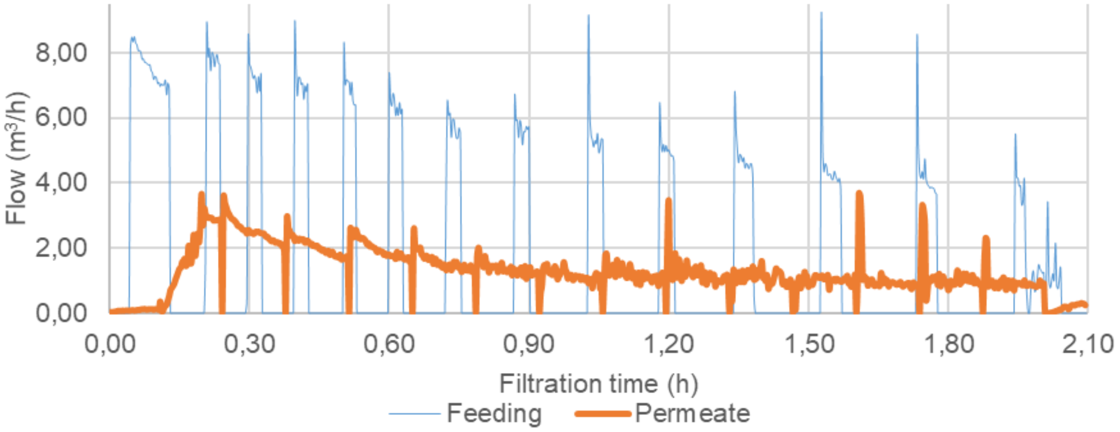

Figure 4.15 – Odontella harvest filtration 10.12.2019.

In comparison filtrating *Odontella* is much faster and easier than filtrating *Nannochloropsis* when the concentration factor is not very high, for example, at 28th of January to filtrate *Nannochloropsis* to a concentration factor of 6.80 took three hours and forty eight minutes and at 10th of December to filtrate *Odontella* to a concentrator of 6.97 took two hours and five minutes, so to concentrate to the almost same factor and with the same conditions (backwash duration, backwash interval of time and TMP) it takes almost twice the time to filtrate *Nannochloropsis* than it takes to filtrate *Odontella*. For a higher concentration factor Is not possible to compared this since there was not a filtration done for Odontella with a concentration factor as high as there was for Nannochloropsis.

In Table 4.8 can be see all the filtrations performed with *Nannochloropsis* supernatant.

Table 4.8 – Supernatant Nannochloropsis harvest filtration results.

Processing Date	Microalgae	Concentration Factor	Average permeate flow (m^3/h)	Time	Backwash (min)	Backwash Duration (s)	Average TMP (bar)	Permeability Check Done Before			Observations
								Permeability Date	Recovery	Class	
13.02.2020	Supernatant *Nannochloropsis*	3,78	2,34	00:34	8	10	-0,40	07.02.2020	46%	IIb	PC→3CS→F
29.05.2020	Supernatant *Nannochloropsis*	3,66	1,83	00:43	8	10	-0,32	28.05.2020a	-	Ib	PC→1CS→F
02.06.2020	Supernatant *Nannochloropsis*	5,73	2,41	01:01	6	10	-0,37	29.05.2020	-	Ia	PC→3CS→WH→20CIP→BW→WH→BW→BW→WH→1CS→F
03.06.2020	Supernatant *Nannochloropsis*	17,71	1,22	07:08	6	10	-0,22	02.06.2020	-	Ia	PC→MB→1CS→F
04.06.2020	Supernatant *Nannochloropsis*	21,07	1,24	08:18	6	10	-0,27	03.06.2020	-	Ia	PC→1CS→F
17.06.2020	Supernatant *Nannochloropsis*	18,97	1,33	07:07	6	10	-0,35	09.06.2020d	-	Ia	PC→8CS→F
18.06.2020	Supernatant *Nannochloropsis*	19,21	1,14	08:15	6	10	-0,39	-	-	-	MB→1CS→F
01.07.2020	Supernatant *Nannochloropsis*	17,42	1,55	05:52	4	10	-0,26	22.06.2020a	-	Ia	PC→9CS→F
02.07.2020	Supernatant *Nannochloropsis*	19,71	1,34	07:49	4	10	-0,32	01.07.2020	-	IIa	PC→1CS→F
13.07.2020	Supernatant *Nannochloropsis*	3,19	3,52	00:23	6	10	-0,39	10.07.2020b	63%	III	PC→3CS→F
29.07.2020	Supernatant *Nannochloropsis*	5,71	1,66	01:28	6	10	-0,20	29.07.2020	91%	IV	PC→F
12.08.2020	Supernatant *Nannochloropsis*	4,69	2,91	00:39	6	10	-0,39	12.08.2020	88%	III	PC→F
17.08.2020	Supernatant *Nannochloropsis*	4,25	2,69	00:38	4	10	-0,38	17.08.2020	83%	III	PC→F
19.08.2020	Supernatant *Nannochloropsis*	6,32	2,41	01:10	6	10	-0,39	19.08.2020	78%	III	PC→F
21.08.2020	Supernatant *Nannochloropsis*	3,06	2,22	00:29	6	10	-0,40	21.08.2020	67%	III	PC→F
24.08.2020	Supernatant *Nannochloropsis*	4,95	2,75	00:46	6	10	-0,39	24.08.2020	66%	III	PC→F
07.09.2020	Supernatant *Nannochloropsis*	4,33	2,63	00:39	6	5	-0,40	07.09.2020	100%	IV	PC→F

From analyzing Table 4.8 we can see that three different backwash intervals of time were tried, eight, six and four minutes. It is clear that a backwash interval of time of six or four minutes is much better than an 8 minutes. For example, the filtration at the 29th of May for a concentration factor of 3.66, with a backwash interval of time of eight minutes, took forty three minutes, as for the filtration at the 12th of August for a concentrator factor of 4.69, with a backwash interval of time of six minutes, took thirty nine minutes and the filtration of the 17th of August for a concentration factor of 4.25, with a backwash interval of time of four minutes, took thirty eight minutes.

Between having a backwash interval of time of six or four minutes will depend on the concentration factor. For a higher concentration factor, as it is the case of the filtration on the 3th of May and the filtration on the 1th of July, a backwash interval of time is much better, as can be seen, for a concentration factor of 17.71 with a backwash interval of time of six minutes, it took seven hours and eight minutes to filtrate and, for a concentration factor of 17.42 with a backwash interval of time of four minutes, it took five hours and fifty two minutes. On the other hand, for a lower concentration factor it will not change much using a backwash interval of time of four or of six minutes. For example on the 17th of August for a concentrator factor 4.25 with a backwash interval of time of four minutes, took thirty eight minutes to filtrate and, on the 7th of September for a concentration factor of 4.33 with a backwash interval of time of six minutes, took thirty nine minutes, so it does not really makes a difference when filtrating for a low concentration factor.

As for the duration of the backwash it can be seen that having a duration of 10 seconds it is better than a duration of 5 seconds. For example, on the 7th of September for a concentration factor of 4.33 with a backwash duration of five seconds, it took thirty nine minutes to filtrate and on the 12th of August for a concentration factor a bit higher, of 4.69, with a backwash duration of ten seconds, also took thirty nine minutes.

As for the highest concentration factor tried was on the 4th of June with a concentration factor of 21.07 and a filtration time of eight hours and eighteen minutes. As can be seen on Figure 4.16 the permeate flow does not goes down a lot throughout the filtration so in terms of concentration this filtration is still viable but, if we look at the time it took to filtrate is longer than the working day's hours (eight hours) so this would leave us with no time to clean the system. So in order to be able to filtrate and then clean the system on the same day, the maximum time a filtration could take was seven hours, leaving one hour for the cleaning steps. Since for this high concentration factor the time in between backwashes should be four minutes and the duration of each backwash should be ten seconds, then there are two filtrations that go according to this, the one on the 1th of July with a concentration factor of 17.42 that took a time of five hours and fifty two minutes, lower than the highest time that is being chosen and, the one on the 2th of July with a concentration factor of 19.71 that took a time seven hours and forty nine minutes, higher that the highest time being considered. So the concentration factor that should be pointed out as to the highest viable concentration factor should be 18 because is in between the two concentration factor and will have more or less the maximum time allowable for the cleaning to also be performed, this will correspond to a processed volume of 8388 L.

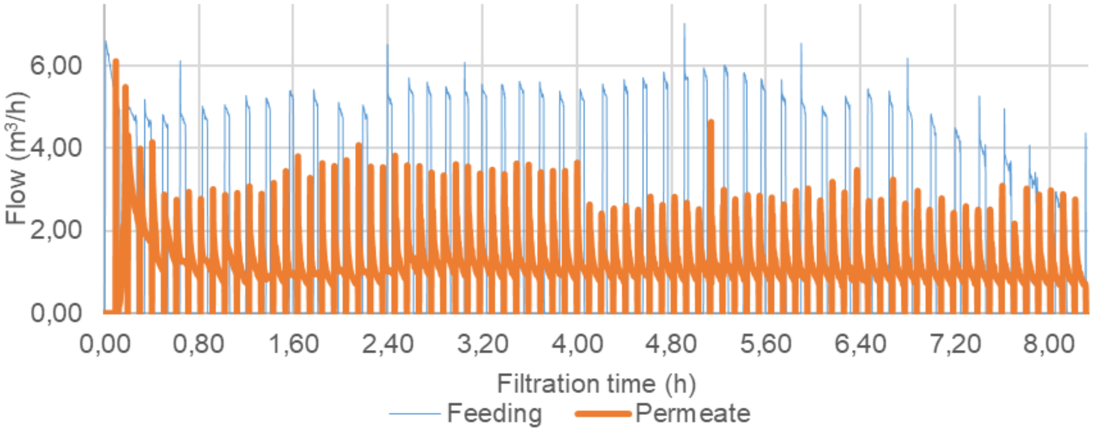

Figure 4.16 – Supernatant Nannochloropsis harvest filtration 04.06.2020.

In Table 4.9 can be seen the two filtrations performed with the supernatant of *Odontella*.

Table 4.9 – Supernatant Odontella harvest filtration results.

Processing Date	Microalgae	Concentration Factor	Average permeate flow (m^3/h)	Time	Backwash (min)	Backwash Duration (s)	Average TMP (bar)	Permeability Check Done Before			Observations
								Permeability Date	Recovery	Class	
05.03.2020	Supernatant *Odontella*	2,67	2,74	00:22	8	10	-0,39	14.02.2020	48%	III	PC→20CS→F
13.08.2020	Supernatant *Odontella*	1,70	3,92	00:15	4	10	-0,37	13.08.2020	69%	III	PC→F

Supernatant of O*dontella* was only filtrated two times, so even though there is two filtrations done with a different time in between backwashes, it is impossible to say which one of the times is more efficient because, on the 5th of March the filtration was performed with a backwash interval of time of eight minutes and the concentration factor was of 2.67, on the other hand the filtration on the 13th of August was performed with a backwash interval of four minutes but the concentration factor was way less, was of 1.70 so it is impossible to compare both.

The backwash duration was impossible to be compared since there was only two filtrations done and the factor that was chosen to be changed was the backwash interval of time.

As to the maximum concentration factor viable, since only two filtrations were performed and both of them with a very low concentration factor then it is impossible to know until what concentration factor the filtration would be viable. The highest concentration factor performed was of 2.67 and, as can be seen in Figure 4.17, the permeate flow almost does not changes from the beginning to the end of the filtration, so until this concentration factor is viable to filtrate but it is impossible to know until what concentration factor is viable.

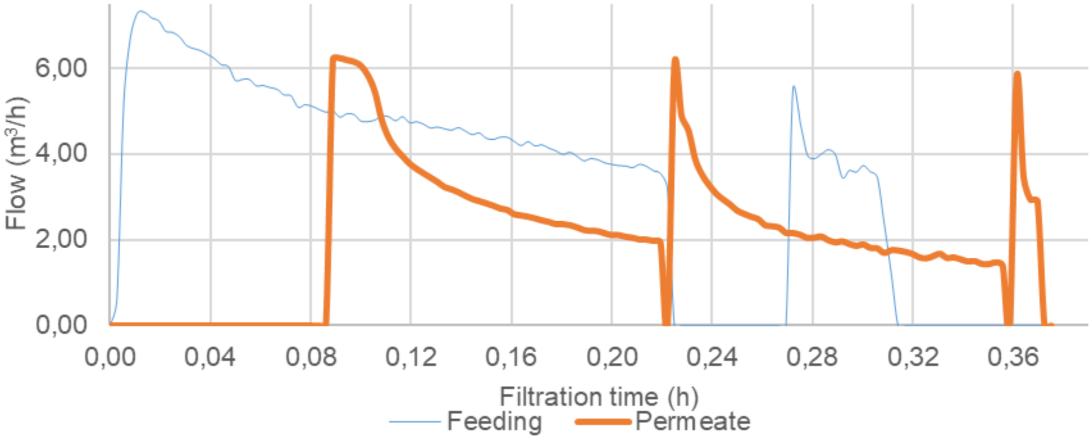

Figure 4.17 – Supernatant Odontella harvest filtration 05.03.2020.

5 Conclusion

The most common steps to clean an ultrafiltration system is using forward flow and backwash, subjecting the membrane to pressure for the dirt to come out, or cleaning it with a basic or acidic solution, depending on the type of fouling the membrane is suffering.

After the tests done with the filtration system the conclusion was that right after a filtration a hose should be passed through the concentrator tank to remove all the foam that is produced when filtrating, then the tank should be filled with water and forward flow and backwash should be performed, always with the blower turned on, in order to clean the membrane to its maximum, this step should be taken until the permeate flow is no longer increasing. Then a permeability check should be performed and the concentrator tank and permeate tank should be left inside a solution of 50 ppm of sodium hypochlorite (common concentration for disinfection used in A4F). On the other day another permeability check should be performed to see if the permeability is lower or higher than 90%, if it is lower than a basic cleaning should be performed and after that a permeability check should be done, if the permeability is still not increasing to 90% or more, than an acidic cleaning should be performed.

The cleaning plan was proved to be efficient throughout the data obtained and seeing that the membrane was clean when it was removed. When the membrane was removed it was seen that it had some bundles torn up so, unless the membrane is replaced, it can never be expected for the system to efficiently filtrate *Nannochloropsis* as this is the smallest microalgae produced by A4F, the other microalgae were never found on the permeate tank so for those it is safe to say the filtration will be efficient.

As for the system itself an option should be created on the program to choose if the concentrated tank should be emptied or not before starting a filtration. A bottom purge should be built on the permeate tank and a water entry should be built in there too. And finally, the pipe that connects the blower to the concentrator tank should be at a higher level than the top of the tank in order to prevent liquid from coming into the blower.

References

[1] M. I. Khan, J. H. Shin, and J. D. Kim, “The promising future of microalgae: Current status, challenges, and optimization of a sustainable and renewable industry for biofuels, feed, and other products,” *Microb. Cell Fact.*, vol. 17, no. 1, pp. 1–21, 2018, doi: 10.1186/s12934-018-0879-x.

[2] P. Spolaore, C. Joannis-Cassan, E. Duran, and A. Isambert, “Commercial applications of microalgae,” *J. Biosci. Bioeng.*, vol. 101, no. 2, pp. 87–96, 2006, doi: 10.1263/jbb.101.87.

[3] N. Sharma and P. Sharma, “JOURNAL OF ADVANCES IN PLANT BIOLOGY Review Industrial and Biotechnological Applications of Algae: A Review,” no. 1, 2017, doi: 10.14302/issn.2638-4469.japb-17-1534.

[4] K. N. C. Murthy, A. Vanitha, J. Rajesha, M. M. Swamy, P. R. Sowmya, and G. A. Ravishankar, “In vivo antioxidant activity of carotenoids from Dunaliella salina - A green microalga,” *Life Sci.*, vol. 76, no. 12, pp. 1381–1390, 2005, doi: 10.1016/j.lfs.2004.10.015.

[5] J. Jia, D. Han, H. G. Gerken, Y. Li, M. Sommerfeld, Q. Hu and J. Xu, “Molecular mechanisms for photosynthetic carbon partitioning into storage neutral lipids in Nannochloropsis oceanica under nitrogen-depletion conditions,” *Algal Res.*, vol. 7, pp. 66–77, 2015, doi: 10.1016/j.algal.2014.11.005.

[6] S. Xia, B. Gao, A. Li, J. Xiong, Z. Ao, and C. Zhang, “Preliminary characterization, antioxidant properties and production of chrysolaminarin from marine diatom Odontella aurita,” *Mar. Drugs*, vol. 12, no. 9, pp. 4883–4897, 2014, doi: 10.3390/md12094883.

[7] H. Elcik and M. Cakmakci, “Harvesting microalgal biomass using crossflow membrane filtration: critical flux, filtration performance, and fouling characterization,” *Environ. Technol. (United Kingdom)*, vol. 38, no. 12, pp. 1585–1596, 2017, doi: 10.1080/09593330.2016.1237560.

[8] I. L. C. Drexler and D. H. Yeh, “Membrane applications for microalgae cultivation and harvesting: a review,” *Rev. Environ. Sci. Biotechnol.*, vol. 13, no. 4, pp. 487–504, 2014, doi: 10.1007/s11157-014-9350-6.

[9] A. Shahid, A. Zafar Khan, T. Liu, S. Malik, I. Afzal, and M. A. Mehmood, *Production and Processing of Algal Biomass*. Elsevier Inc., 2017.

[10] G. Singh and S. K. Patidar, “Microalgae harvesting techniques: A review,” *J. Environ. Manage.*, vol. 217, pp. 499–508, 2018, doi: 10.1016/j.jenvman.2018.04.010.

[11] Rismiati, “Study of Filtration Characteristics of Crossflow Filtration for Cable Suspended Robot - Algae Harvester,” no. August, 2016, [Online]. Available: https://www.semanticscholar.org/paper/Study-of-Filtration-Characteristics-of-Crossflow-Karisiddappa/7138641c226dde8ab48c231401915ea071d6e732.

[12] M. R. Bilad, V. Discart, D. Vandamme, I. Foubert, K. Muylaert, and I. F. J. Vankelecom, “Harvesting microalgal biomass using a magnetically induced membrane vibration (MMV) system: Filtration performance and energy consumption,” *Bioresour. Technol.*, vol. 138, pp. 329–338, 2013, doi: 10.1016/j.biortech.2013.03.175.

[13] Z. Zhao, Y. Li, K. Muylaert, and I. F. J. Vankelecom, “Synergy between membrane filtration and flocculation for harvesting microalgae,” *Sep. Purif. Technol.*, vol. 240, no. November 2019, p. 116603, 2020, doi: 10.1016/j.seppur.2020.116603.

[14] F. Zhao, Z. Li, X. Han, X. Zhou, Y. Zhang, S. Jiang, Z. Yu, X. Zhou, C. Liu and H. Chu, “The interaction between microalgae and membrane surface in filtration by uniform shearing vibration membrane,” *Algal Res.*, vol. 50, no. April, p. 102012, 2020, doi: 10.1016/j.algal.2020.102012.

A. [15] A. K. S. Lau, M. R. Bilad, N. A. H. M. Nordin, K. Faungnawakij, T. Narkkun, D. K. Wang, T. M. I. Mahlia and J. Jaafar, “Effect of membrane properties on tilted panel performance of microalgae biomass filtration for biofuel feedstock," *Renew. Sustain. Energy Rev.*, vol. 120, no. November 2019, p. 109666, 2020, doi: 10.1016/j.rser.2019.109666.

[16] E. D. Munz, "Membrane filtration handbook practical tips and hints," *Nervenheilkunde*, vol. 36, no. 10, pp. 800–805, 2001, doi: 10.1007/s13398-014-0173-7.2.

[17] S. Kang, S. Kim, and J. Lee, "Optimization of cross flow filtration system for Dunaliella tertiolecta and Tetraselmis sp. microalgae harvest," *Korean J. Chem. Eng.*, vol. 32, no. 7, pp. 1377–1380, 2015, doi: 10.1007/s11814-014-0343-5.

[18] R. W. Baker, "Ultrafiltration," *Membr. Technol. Appl.*, vol. Third Edit, 2012.

[19] D. M. Stevens, M. L. Stone, E. S. Peterson, and D. T. Newby, "Cross-Flow Filtration of Multiple Algal Strains and Mixed Populations Using Embedded Membranes," no. April, p. 16, 2013, [Online]. Available: https://inldigitallibrary.inl.gov/STI/5753428.pdf.

[20] M. Rickman, J. Pellegrino, and R. Davis, "Fouling phenomena during membrane filtration of microalgae," *J. Memb. Sci.*, vol. 423–424, pp. 33–42, 2012, doi: 10.1016/j.memsci.2012.07.013.

[21] H. Li and V. Chen, *Membrane Fouling and Cleaning in Food and Bioprocessing*, First Edit. Elsevier Ltd, 2010.

[22] "Four types of Membrane Fouling - Membrane Solutions," 2020. https://www.membrane-solutions.com/News_1224.htm (accessed Apr. 20, 2020).

[23] "What Are the Different Types of Membrane Fouling and What Causes Them?" https://www.samcotech.com/types-of-membrane-fouling-and-causes/ (accessed Apr. 20, 2020).

[24] N. Porcelli and S. Judd, "Chemical cleaning of potable water membranes: A review" *Sep. Purif. Technol.*, vol. 71, no. 2, pp. 137–143, 2010, doi: 10.1016/j.seppur.2009.12.007.

[25] S. Adham, K. Chiu and G. Lehman, "Optimization of Membrane Treatment for Direct and Clarified Water Filtration" AwwaRF, 2007

[26] G. Pearce, T. Stepheson, G. Daigger, B. Verrecht, E. Germain, and G. Hill, "Membrane Fundamentals," in *The Membrane Bioreactor Book*, 2011, pp. 55–207.

[27] T. Ref, P. Intel, T. Refundido, P. Intelectual, S. Consolidated, and C. Act, "OPTIMIZATION OF ULTRAFILTRATION MEMBRANE CLEANING PROCESSES . PRETREATMENT FOR REVERSE OSMOSIS IN SEAWATER Optimization of ultrafiltration membrane cleaning processes Pretreatm ent for rev erse osm osis in seaw ater d esalination plants Guillem Gilabert O," 2013.

[28] I. KONKOLY THEGE, "ChemInform Abstract: DSC Investigation of the Thermal Behaviour of (NH 4) 2 SO 4 , NH 4 HSO 4 and NH 4 NH 2 · SO 3 .," *Chem. Informationsd.*, vol. 14, no. 14, pp. 5–6, 1983, doi: 10.1002/chin.198314019.

[29] Australian Government Office of the Gene Technology Regulator, "The Biology of Nannochloropsis oceanica Suda & Miyashita (a microalga)," no. October, 2019, Accessed: Sep. 27, 2020. [Online]. Available: http://webcache.googleusercontent.com/search?q=cache:6VHNztijO8YJ:www.ogtr.gov.au/internet/ogtr/publishing.nsf/Content/5DCF28AD2F3779C4CA257D4E001819B9/%24File/Biology%2520of%2520Nannochloropsis%2520oceanica%2520(a%2520microalga).pdf+&cd=6&hl=en&ct=clnk&g.

[30] "Odontella aurita (Lyngbye) C.A. Agardh, 1832 | Nordic Microalgae." http://nordicmicroalgae.org/taxon/Odontella aurita (accessed Sep. 27, 2020).

[31] A. Hosseini Tafreshi and M. Shariati, "Dunaliella biotechnology: Methods and applications," *Journal of Applied Microbiology*, vol. 107, no. 1. John Wiley & Sons, Ltd, pp. 14–35, Jul. 01, 2009, doi: 10.1111/j.1365-2672.2009.04153.x.

APPENDIX

Appendix A – Permeability check graphics

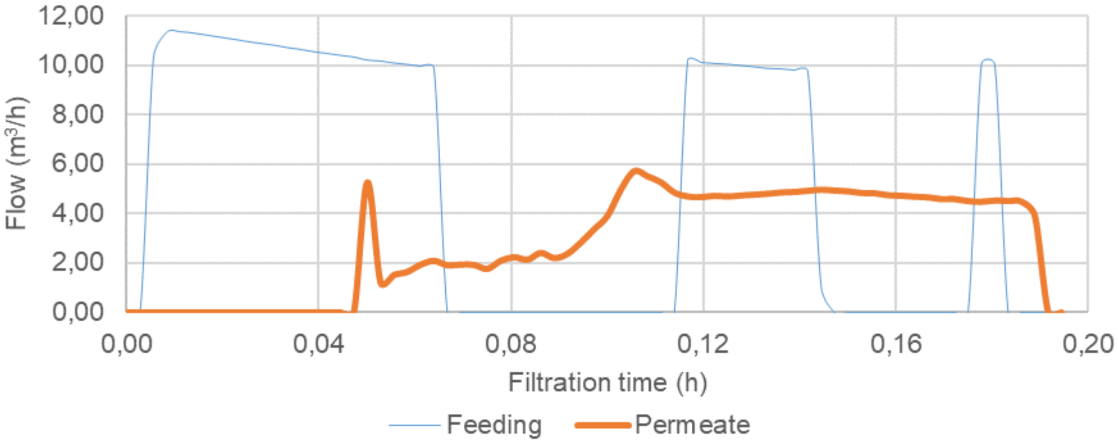

Figure A.1 – Permeability check 06.09.2019.

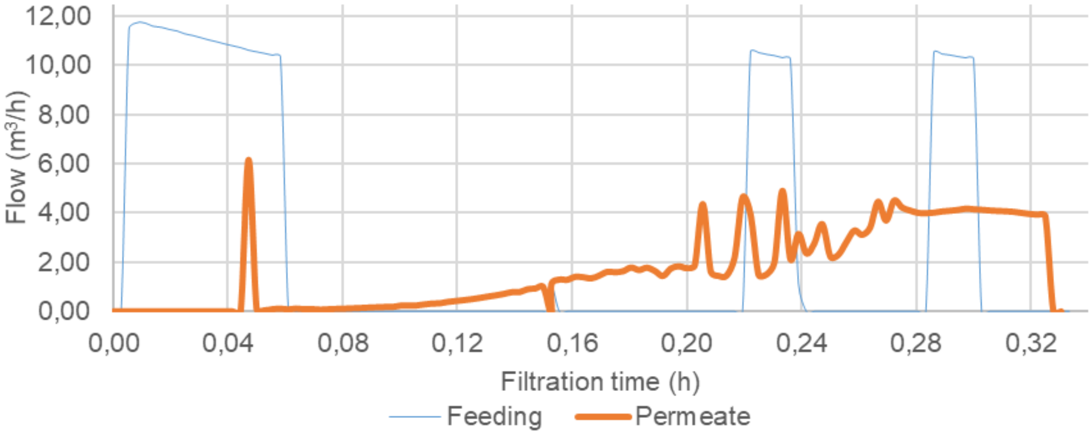

Figure A.2 – Permeability check 10.09.2019.

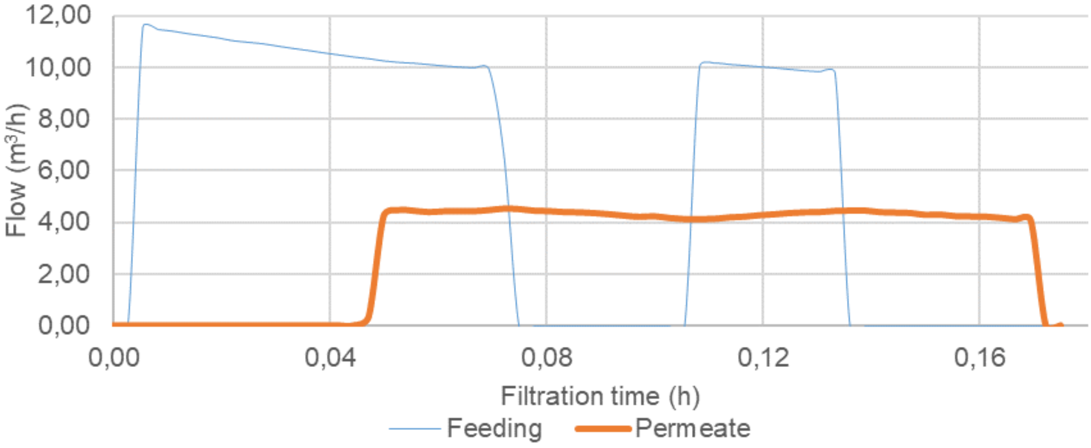

Figure A.3 – Permeability check 10.09.2019a.

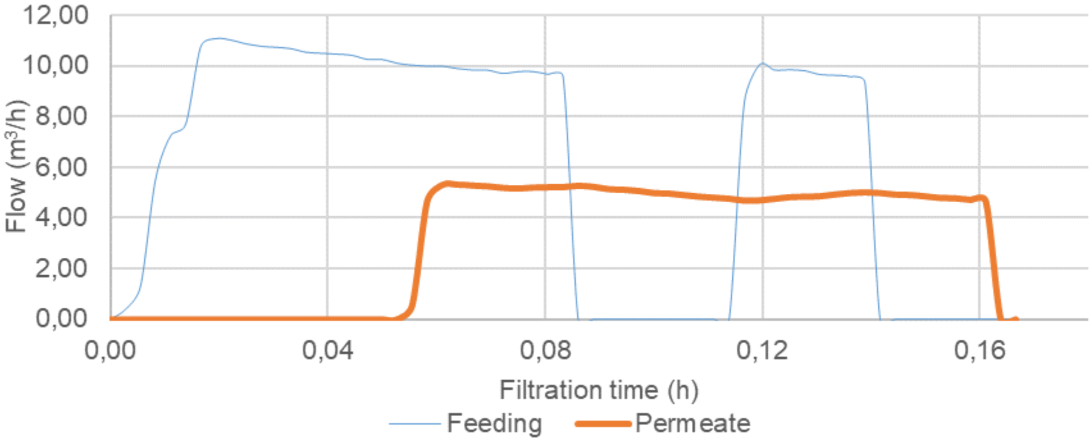

Figure A.4 – Permeability check 13.09.2019.

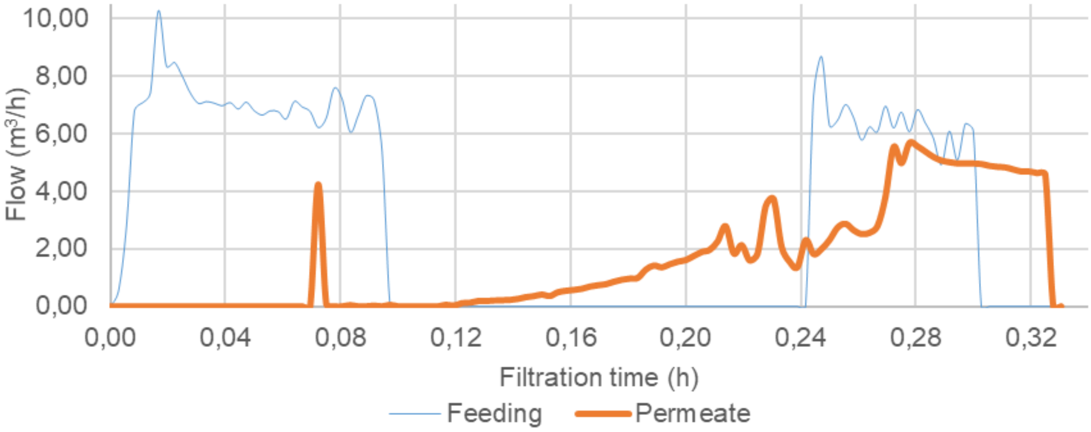

Figure A.5 – Permeability check 30.09.2019.

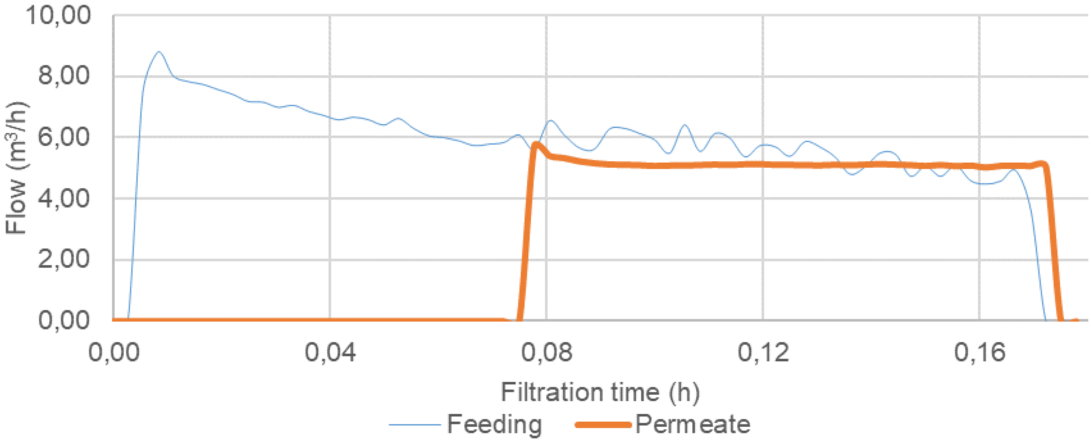

Figure A.6 – Permeability check 30.09.2019a.

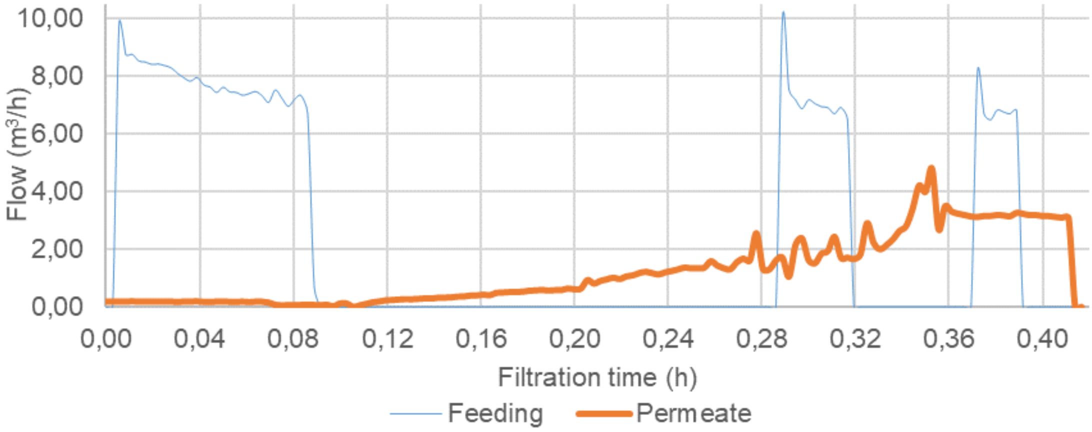

Figure A.7 – Permeability check 29.10.2019.

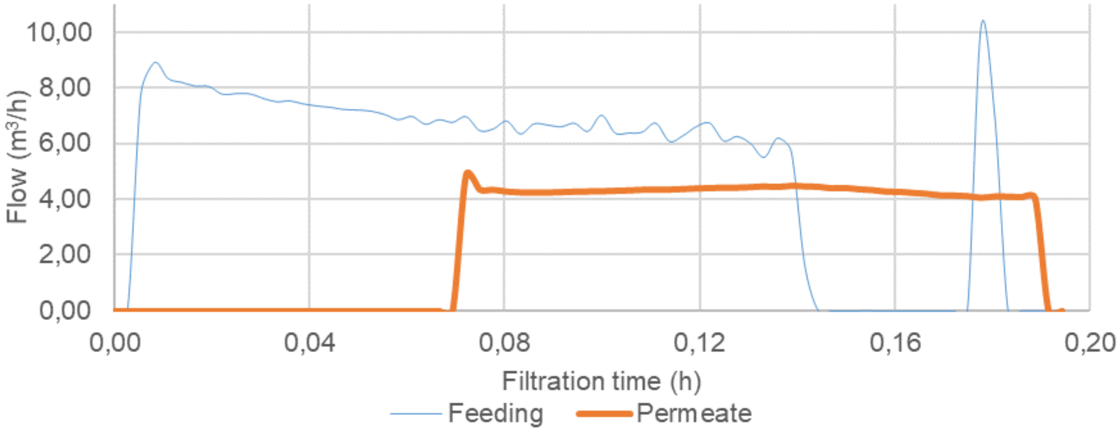

Figure A.8 – Permeability check 30.10.2019.

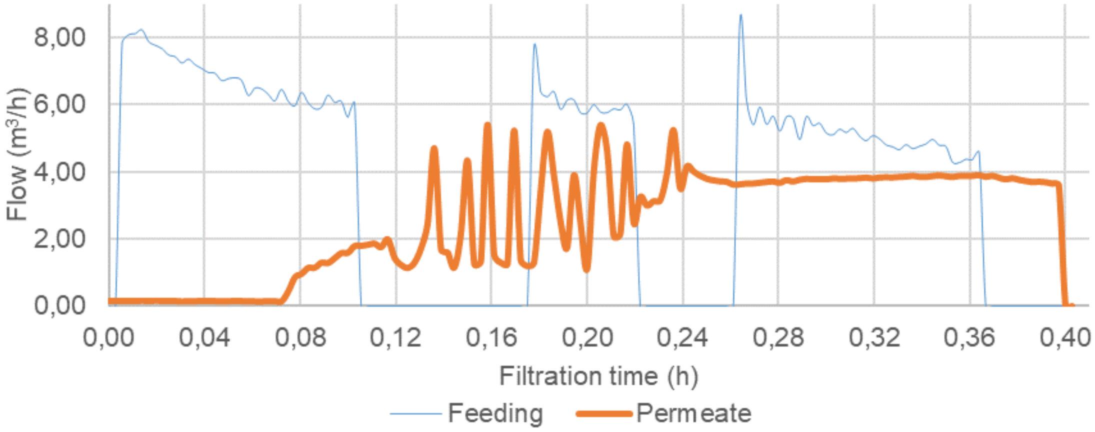

Figure A.9 – Permeability check 13.12.2019.

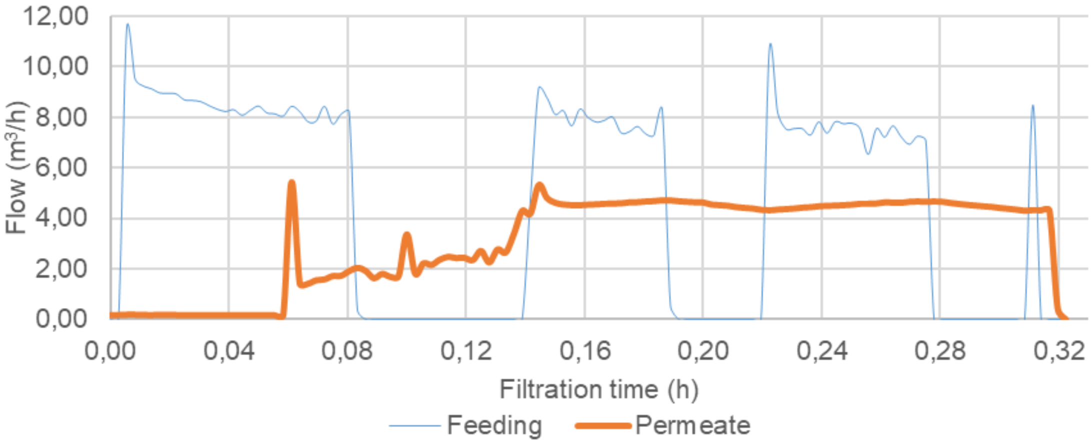

Figure A.10 – Permeability check 16.12.2019.

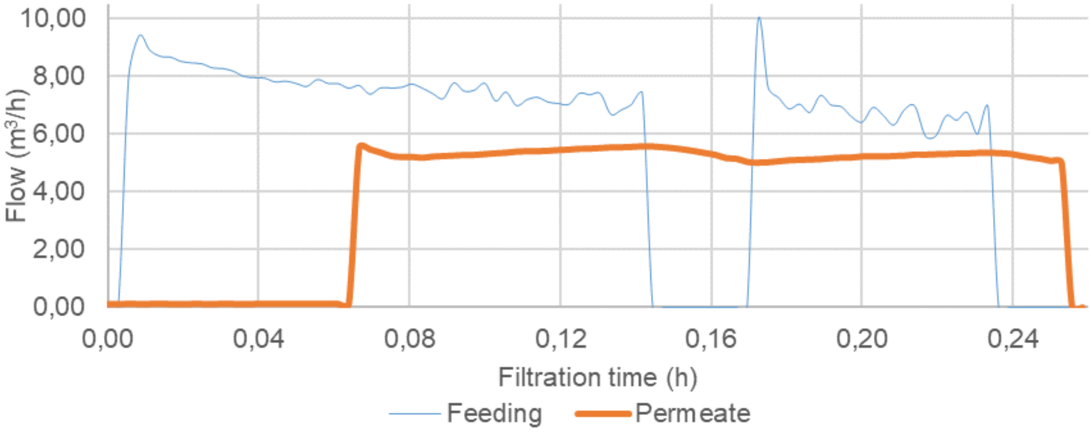

Figure A.11 – Permeability check 19.12.2019.

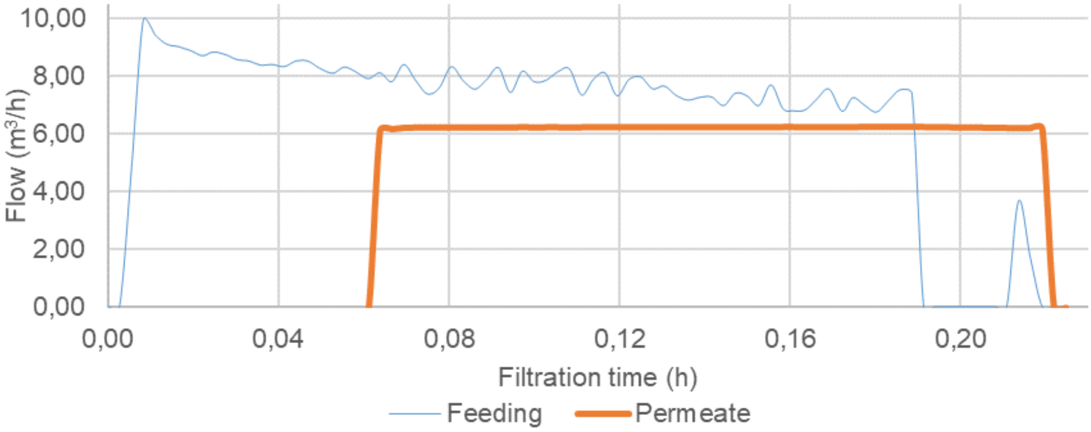

Figure A.12 – Permeability check 30.12.2019.

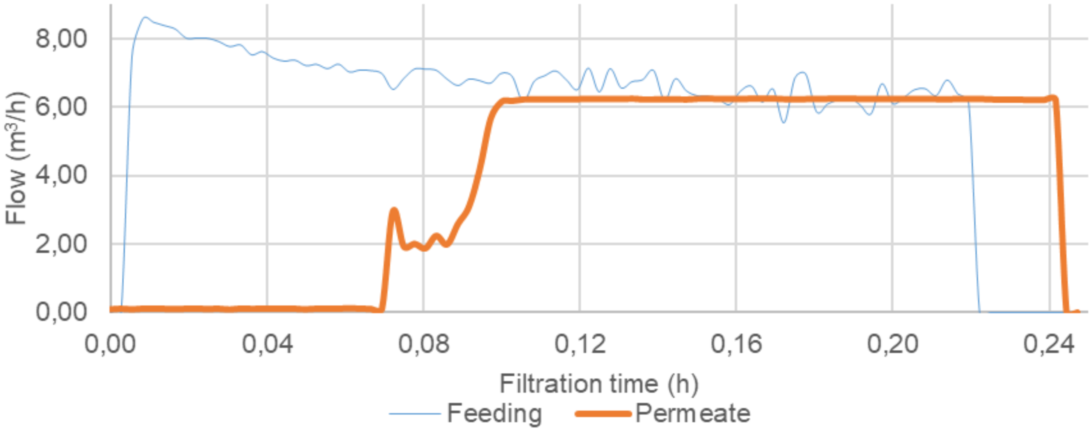

Figure A.13 – Permeability check 06.01.2020.

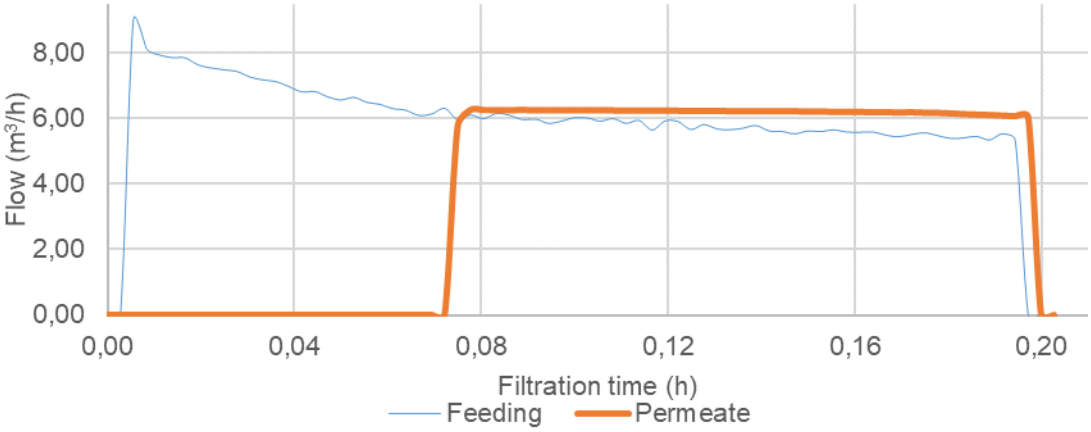

Figure A.14 – Permeability check 06.01.2020a.

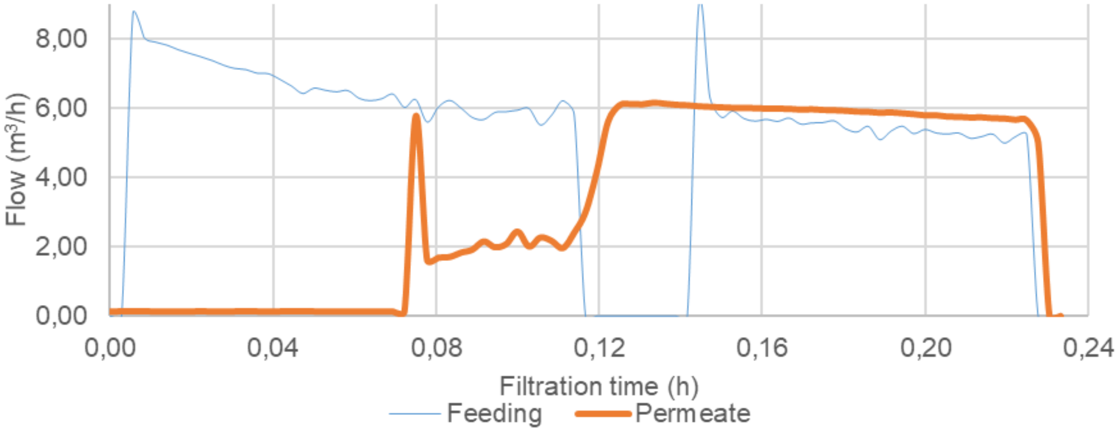

Figure A.15 – Permeability check 10.01.2020.

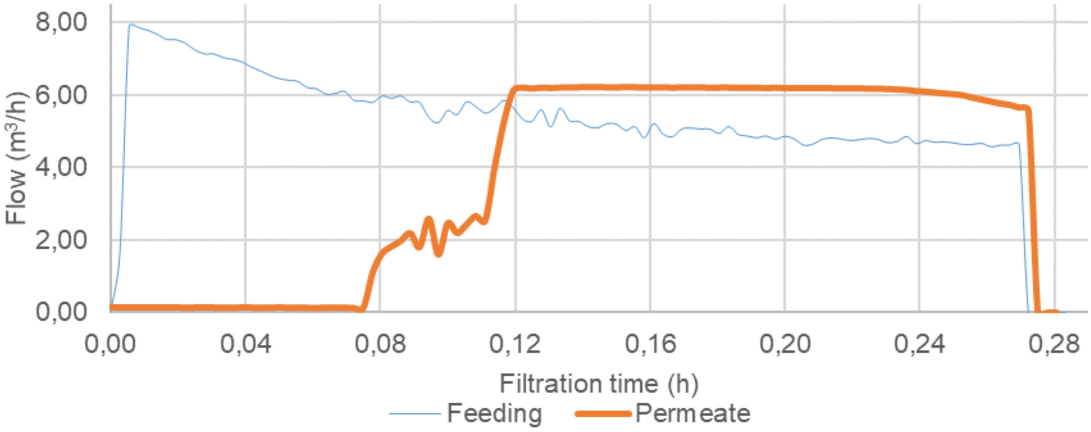

Figure A.16 – Permeability check 17.01.2020.

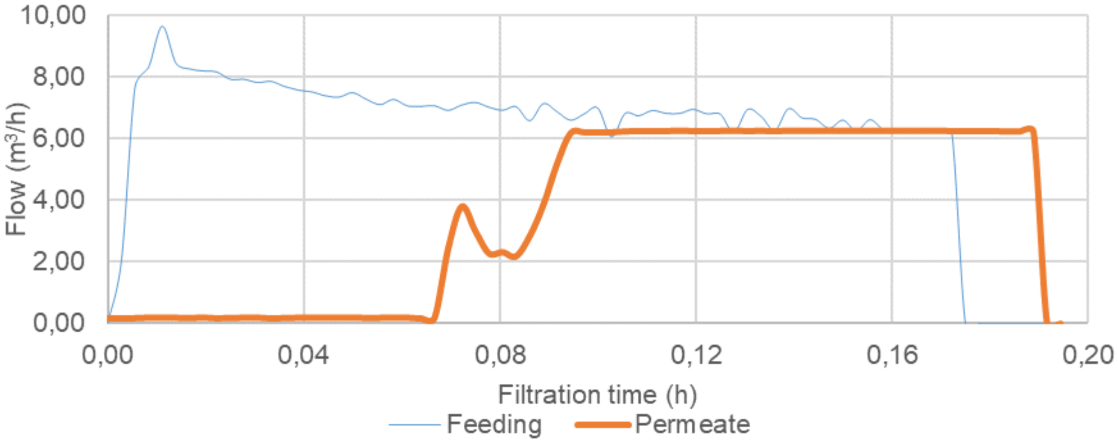

Figure A.17 – Permeability check 21.01.2020.

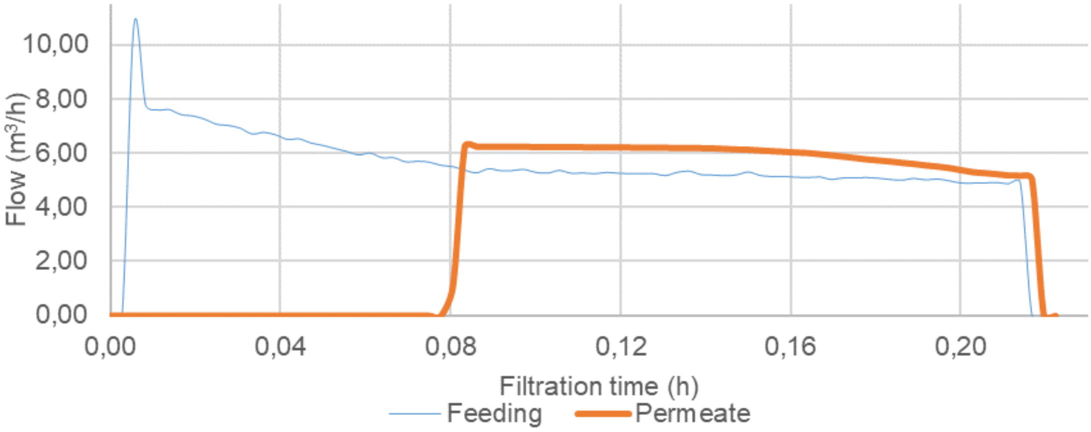

Figure A.18 – Permeability check 21.01.2020a.

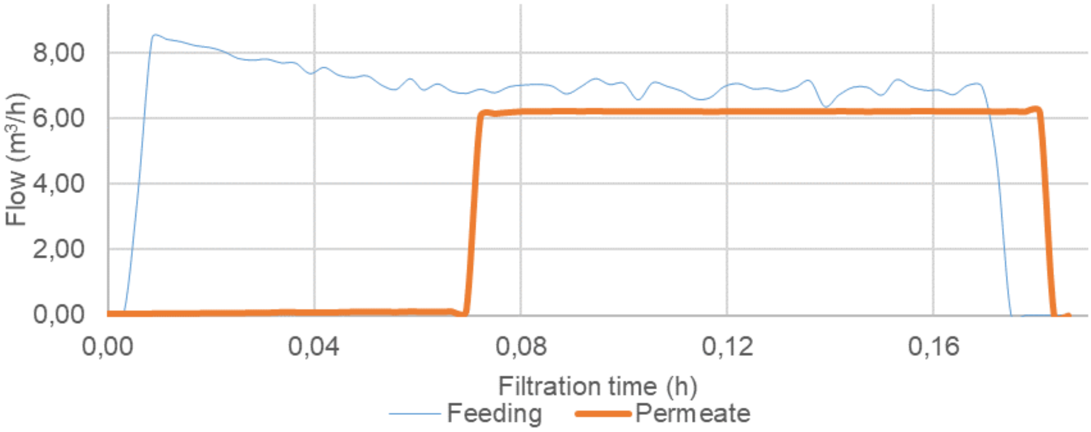

Figure A.19 – Permeability check 22.01.2020.

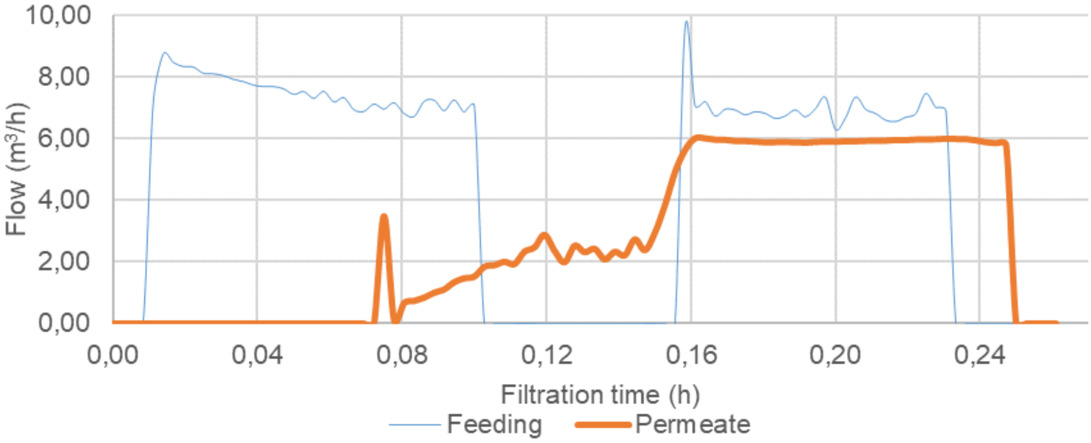

Figure A.20 – Permeability check 24.01.2020.

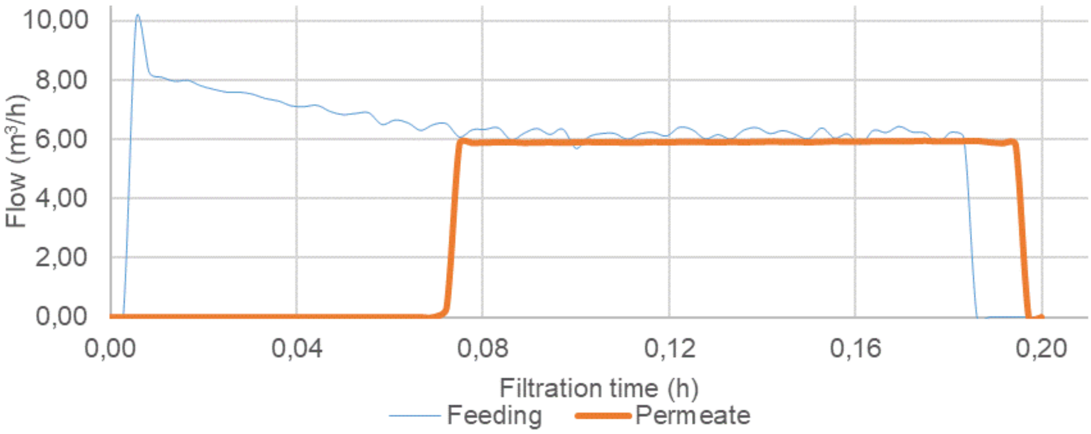

Figure A.21 – Permeability check 24.01.2020a.

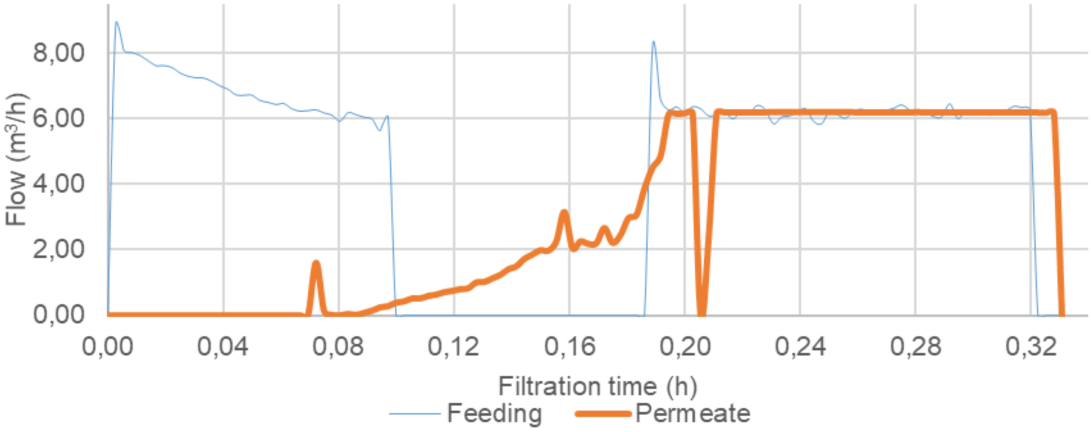

Figure A.22 – Permeability check 30.01.2020.

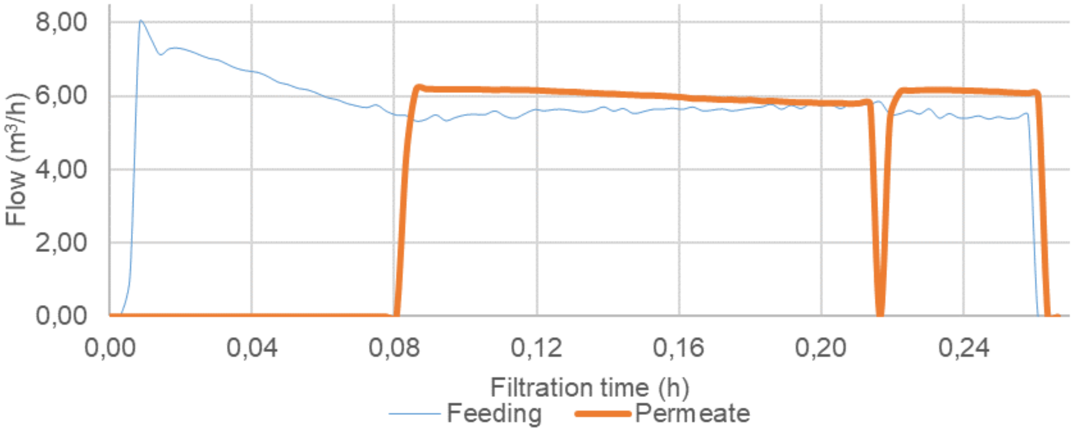

Figure A.23 – Permeability check 30.01.2020a.

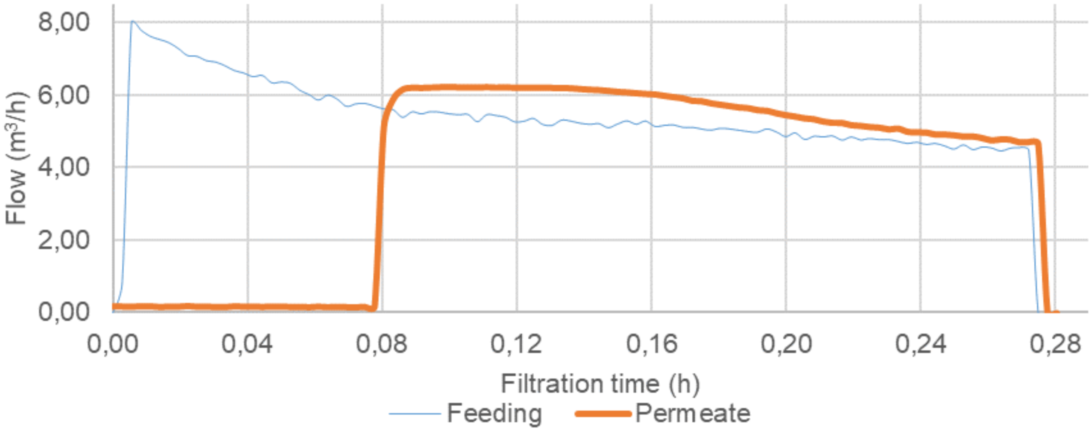

Figure A.24 – Permeability check 31.01.2020.

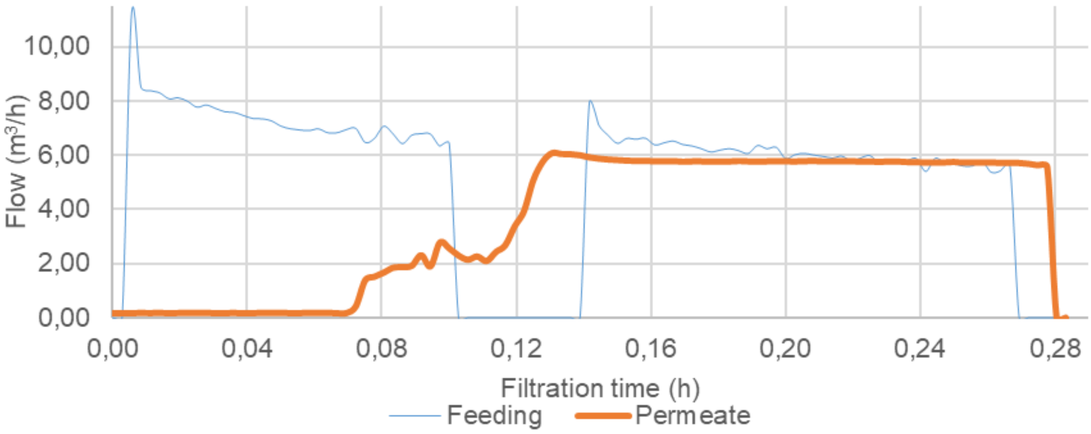

Figure A.25 – Permeability check 07.02.2020.

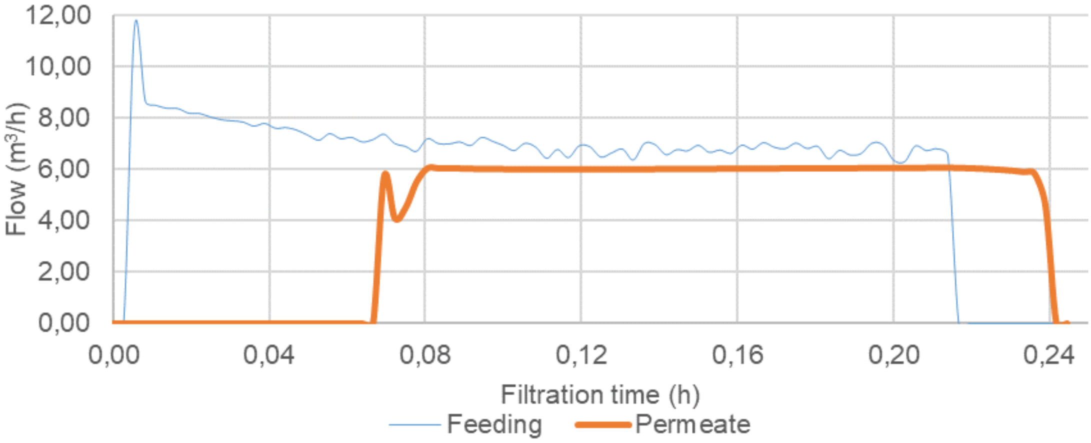

Figure A.26 – Permeability check 14.02.2020.

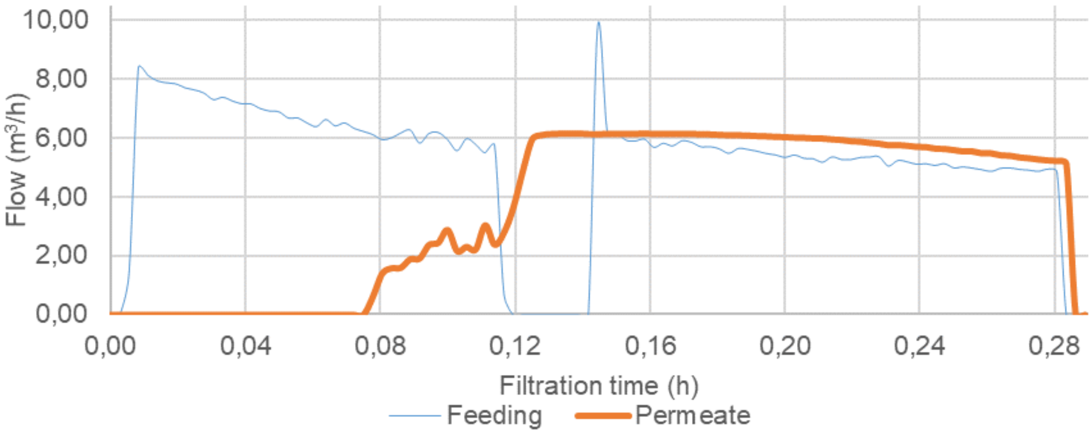

Figure A.27 – Permeability check 06.03.2020.

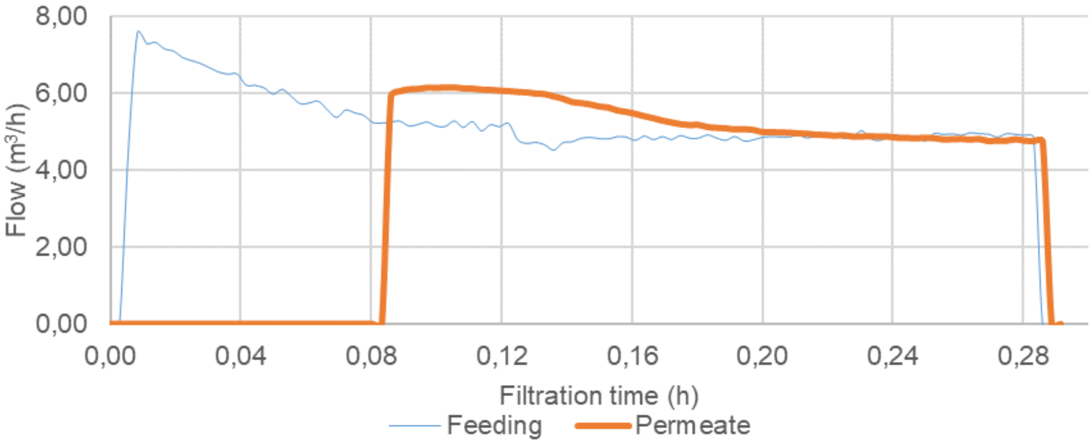

Figure A.28 – Permeability check 06.03.2020a.

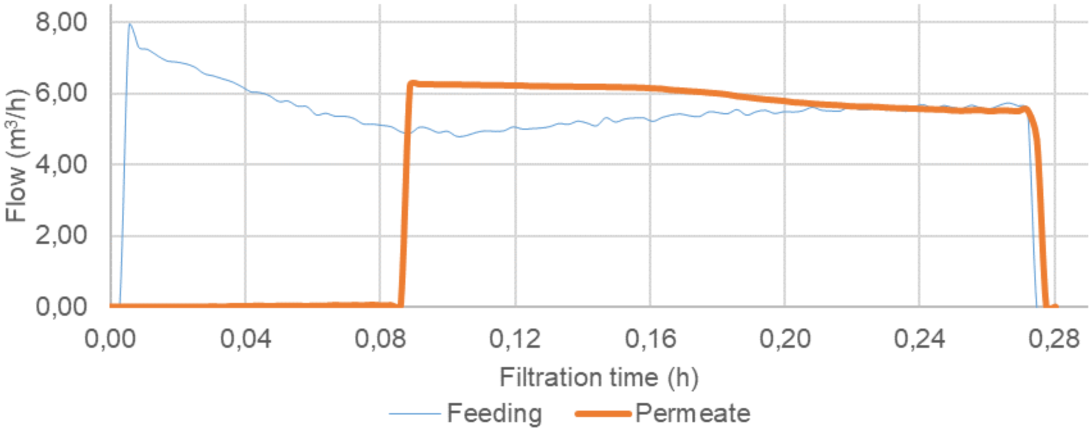

Figure A.29 – Permeability check 11.03.2020.

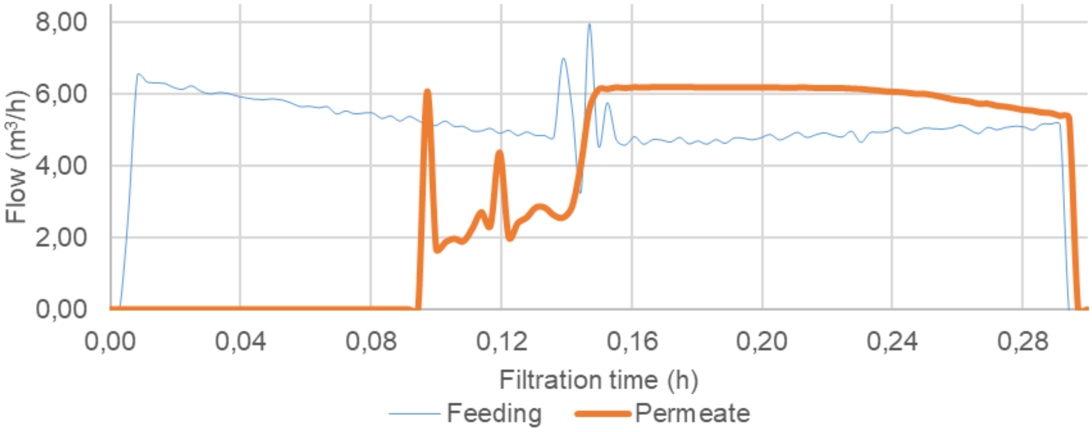

Figure A.30 – Permeability check 12.03.2020.

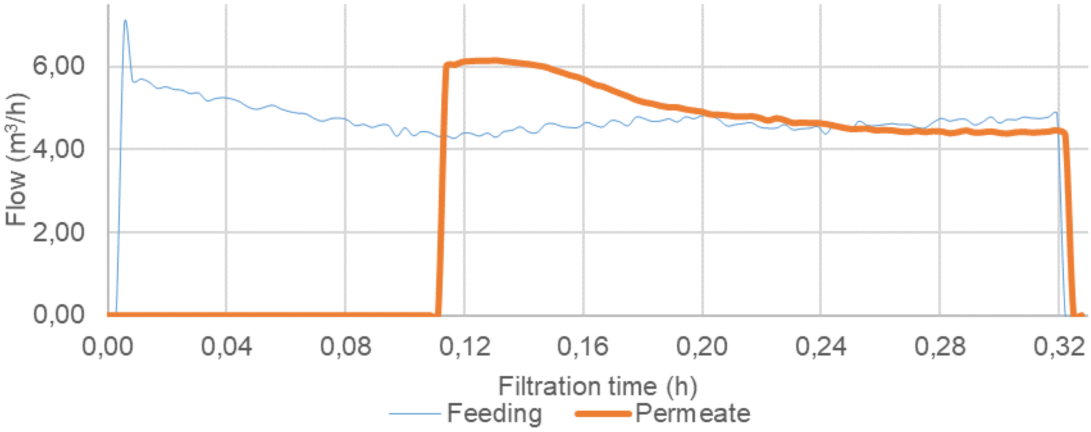

Figure A.31 – Permeability check 12.03.2020a.

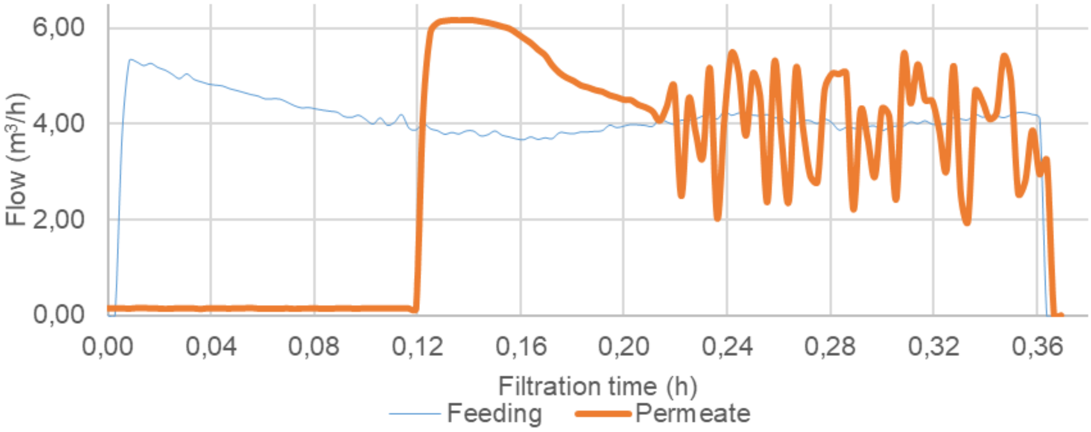

Figure A.32 – Permeability check 13.03.2020.

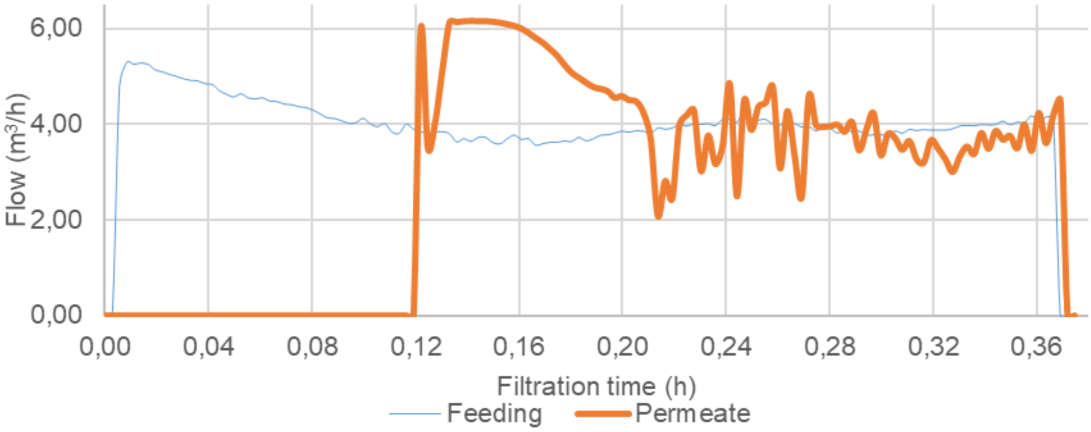

Figure A.33 – Permeability check 13.03.2020a.

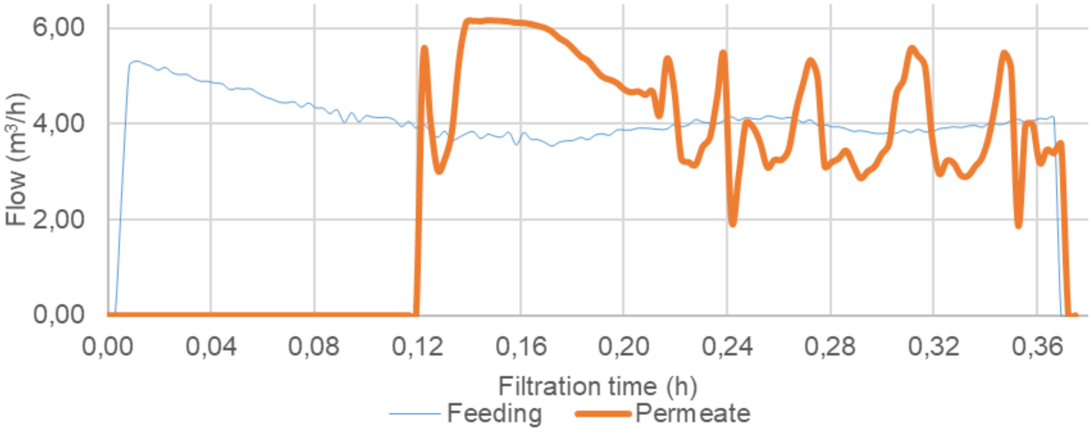

Figure A.34 – Permeability check 13.03.2020b.

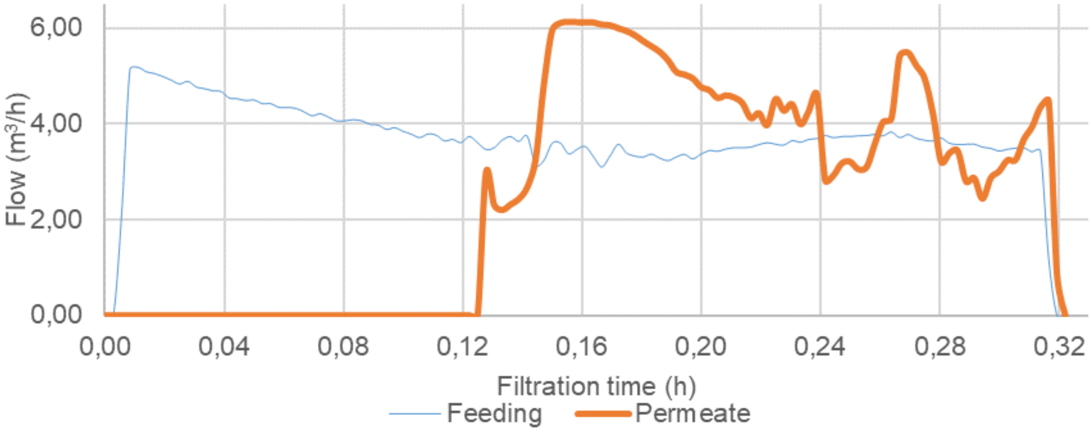

Figure A.35 – Permeability check 16.03.2020.

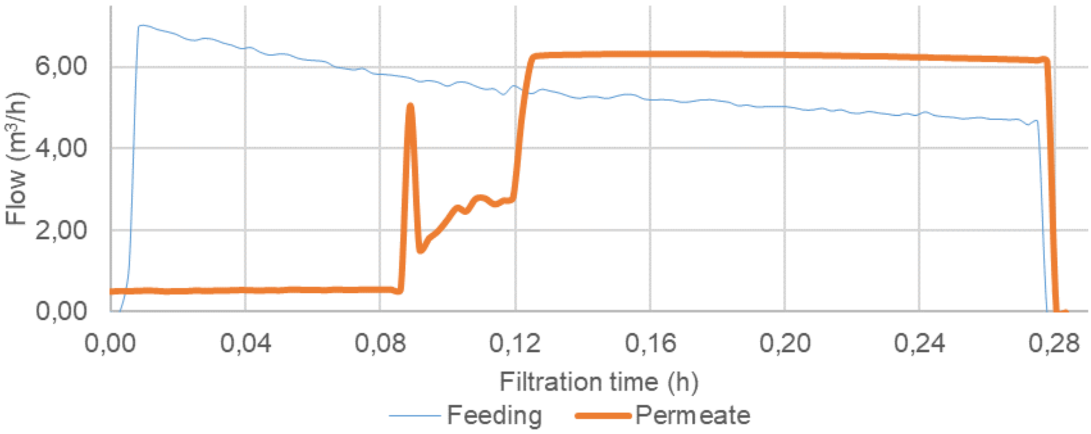

Figure A.36 – Permeability check 28.05.2020.

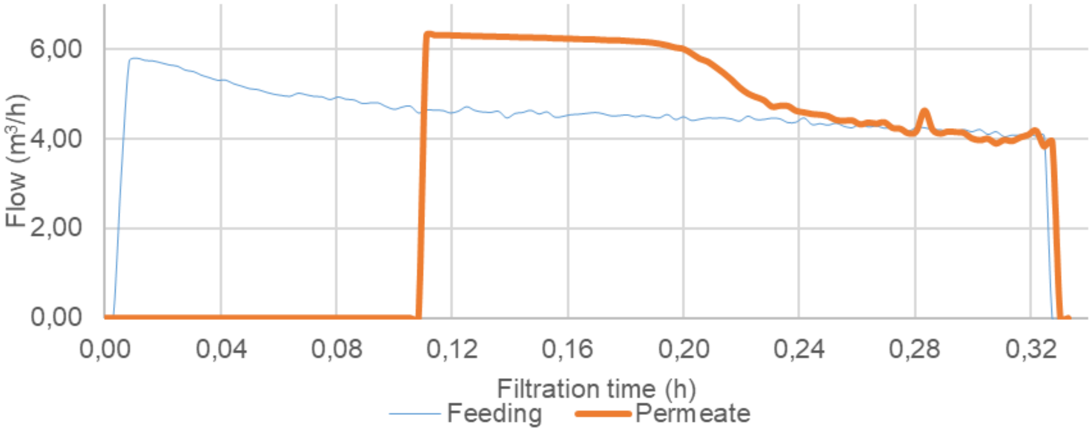

Figure A.37 – Permeability check 28.05.2020a.

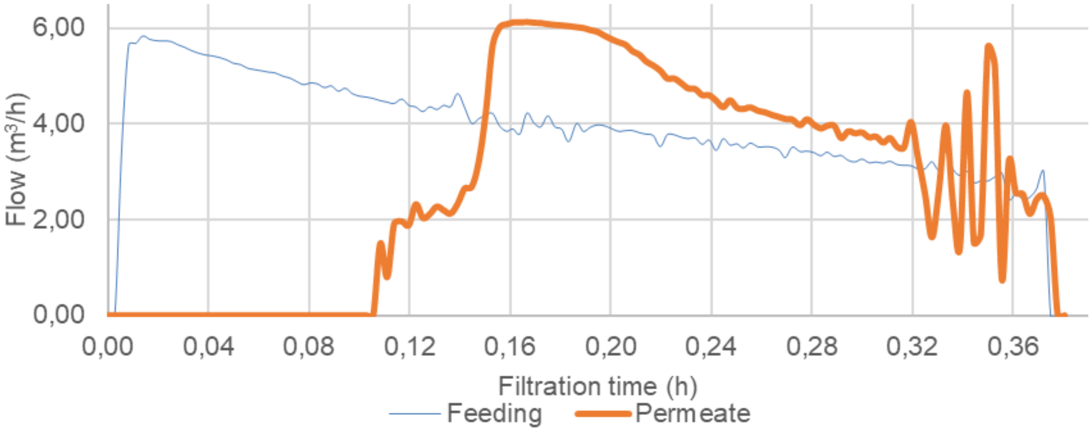

Figure A.38 – Permeability check 29.05.2020.

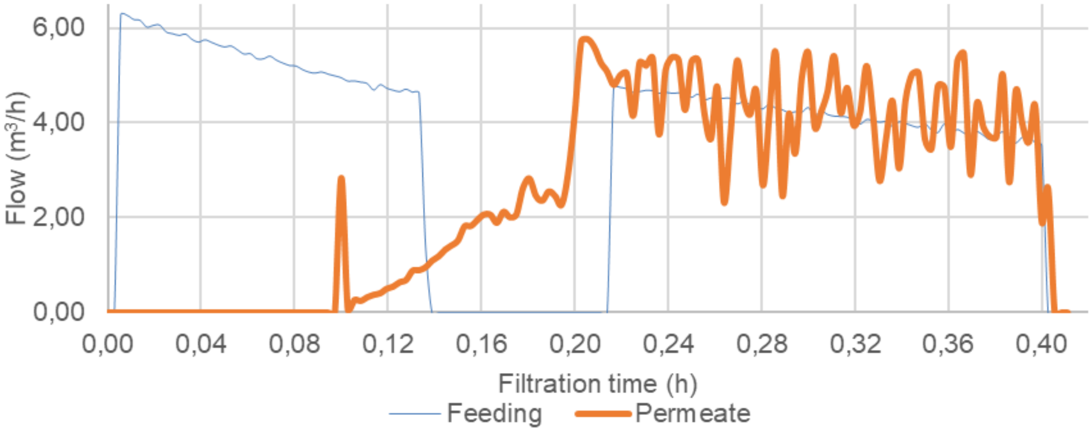

Figure A.39 – Permeability check 02.06.2020.

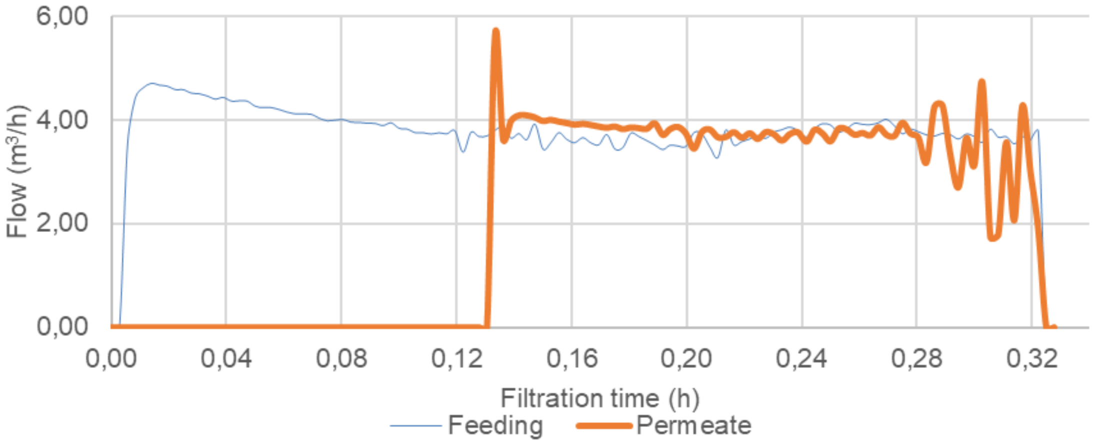

Figure A.40 – Permeability check 03.06.2020.

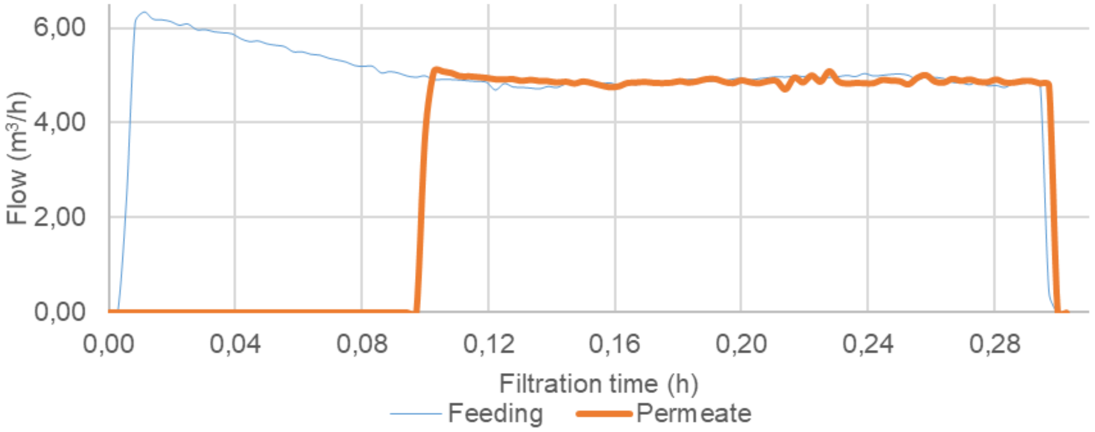

Figure A.41 – Permeability check 05.06.2020.

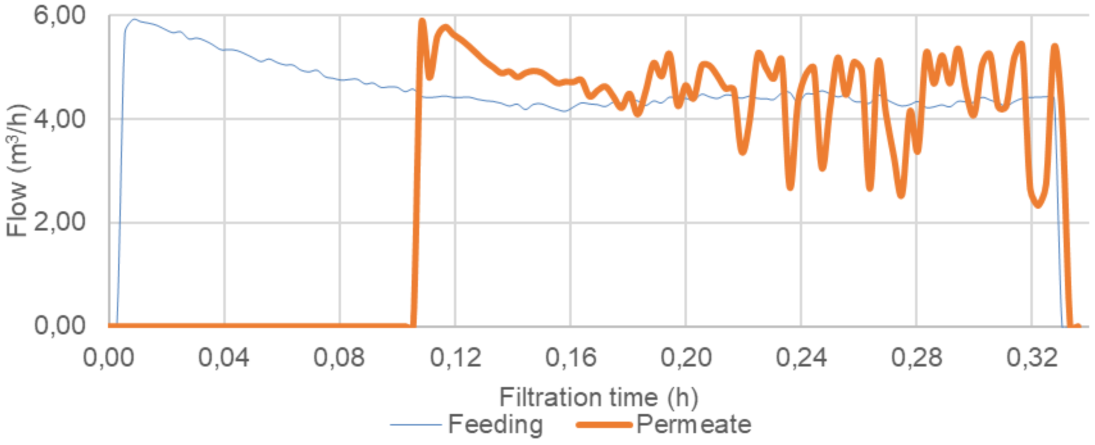

Figure A.42 – Permeability check 05.06.2020a.

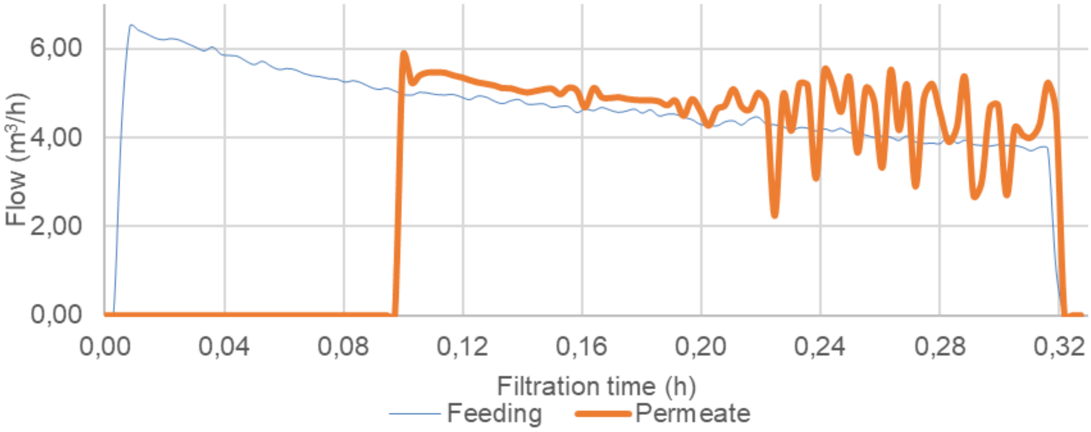

Figure A.43 – Permeability check 05.06.2020b.

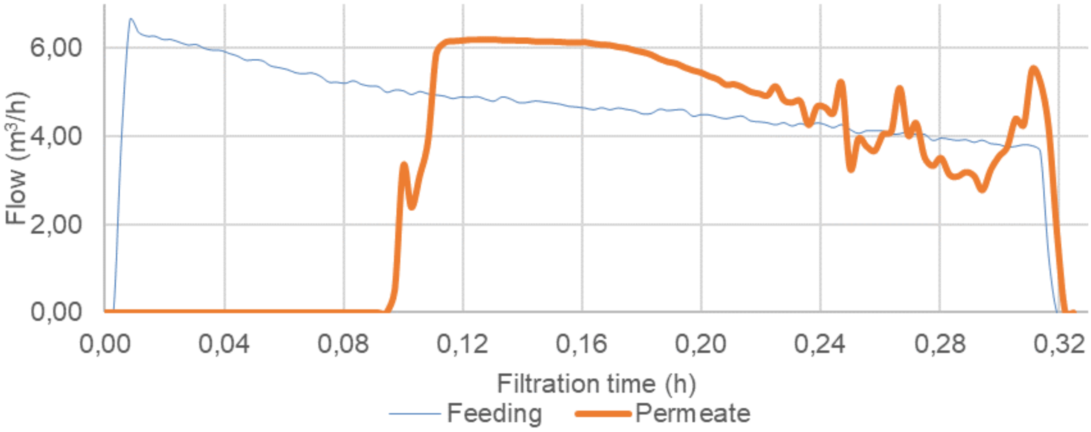

Figure A.44 – Permeability check 09.06.2020.

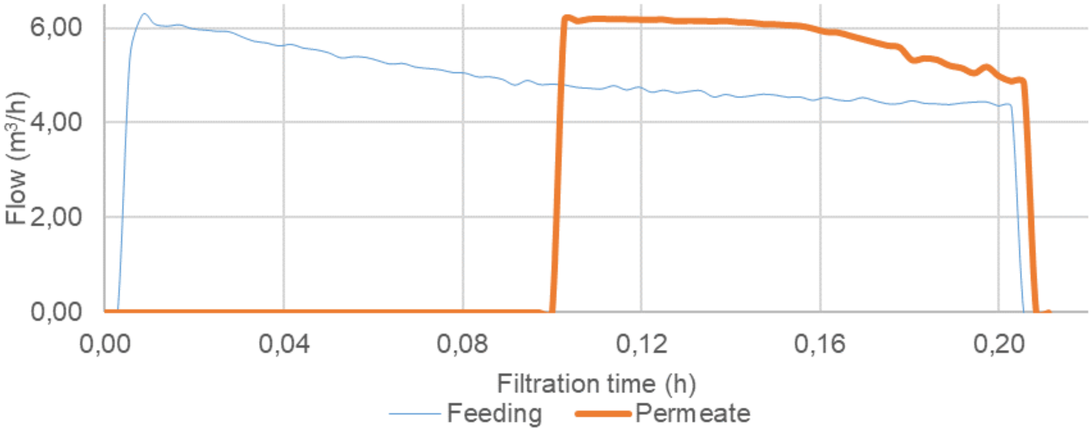

Figure A.45 – Permeability check 09.06.2020a.

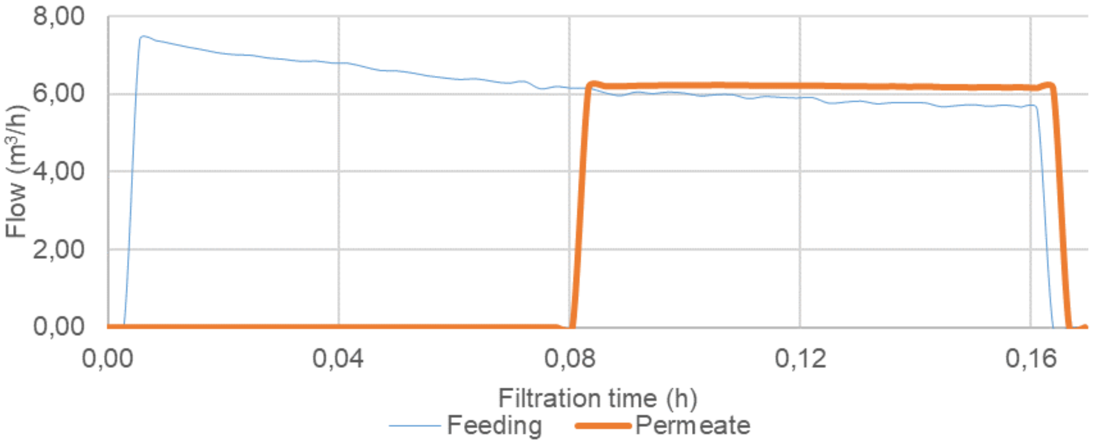

Figure A.46 – Permeability check 09.06.2020b.

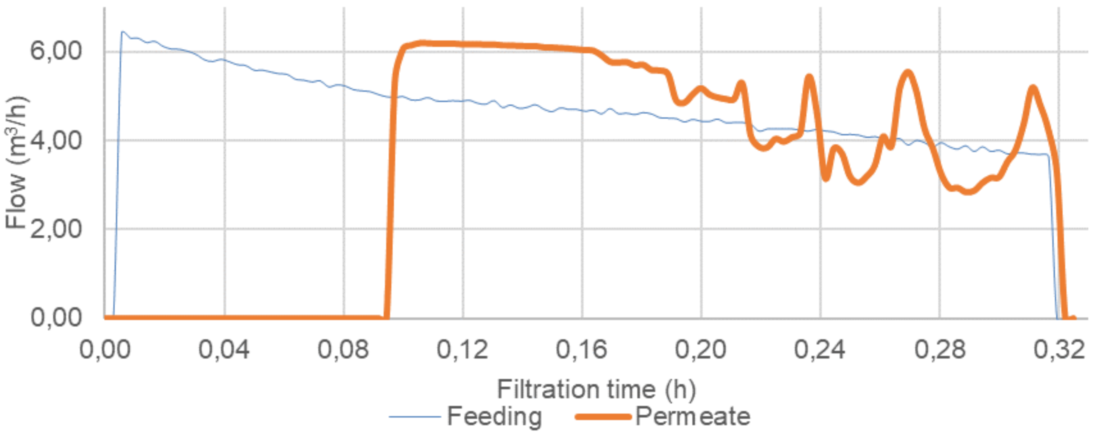

Figure A.47 – Permeability check 09.06.2020c.

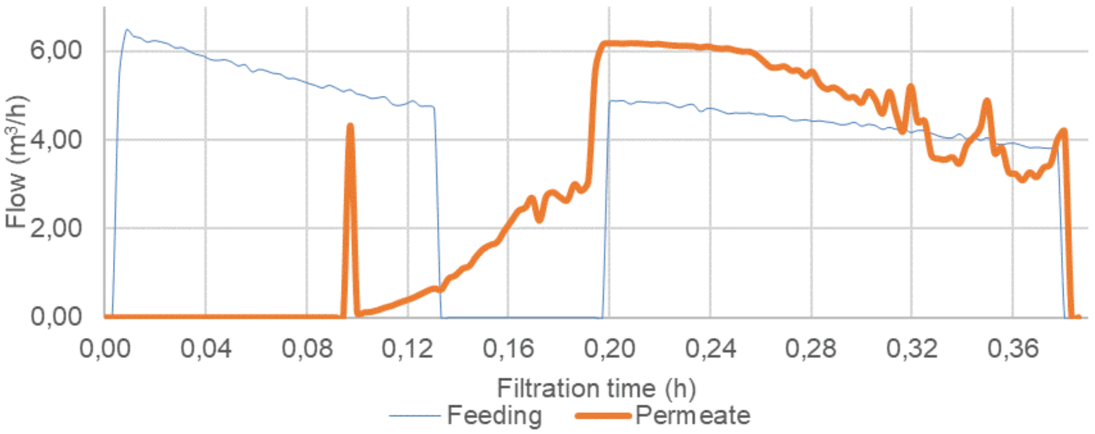

Figure A.48 – Permeability check 09.06.2020d.

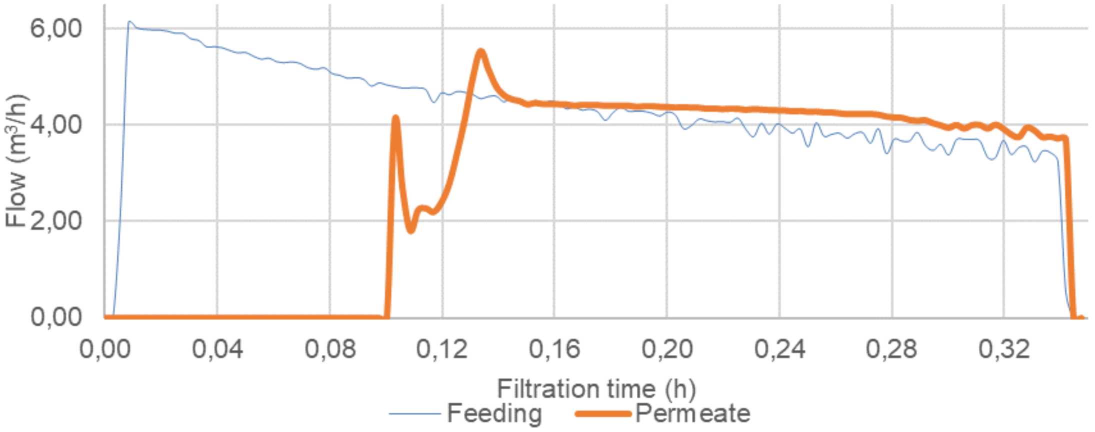

Figure A.49 – Permeability check 22.06.2020.

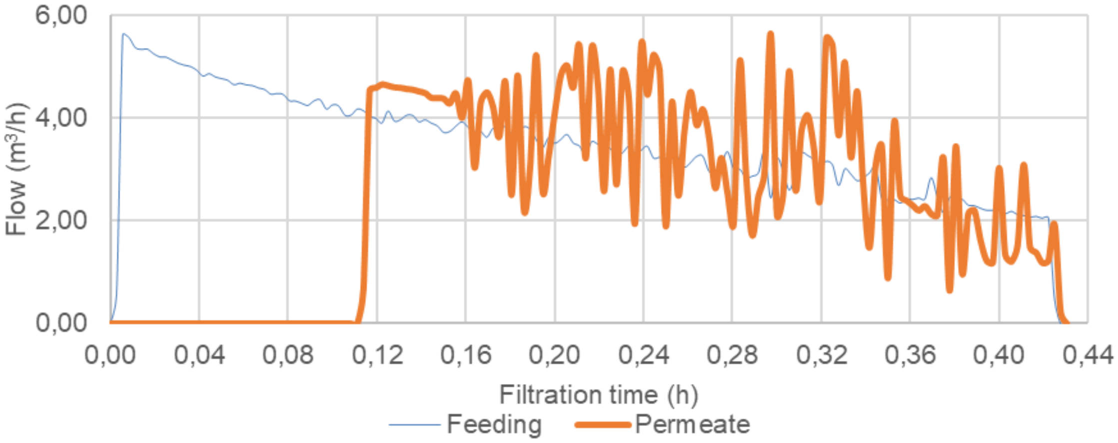

Figure A.50 – Permeability check 22.06.2020a.

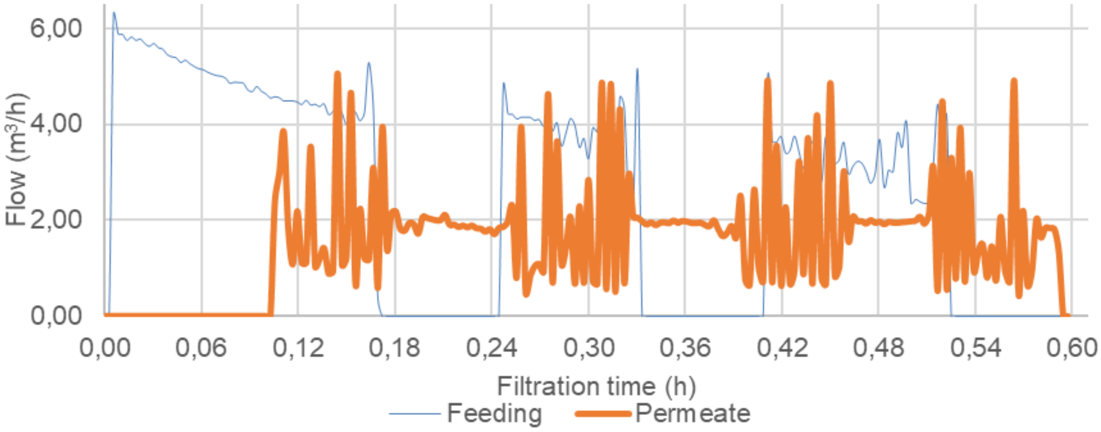

Figure A.51 – Permeability check 01.07.2020.

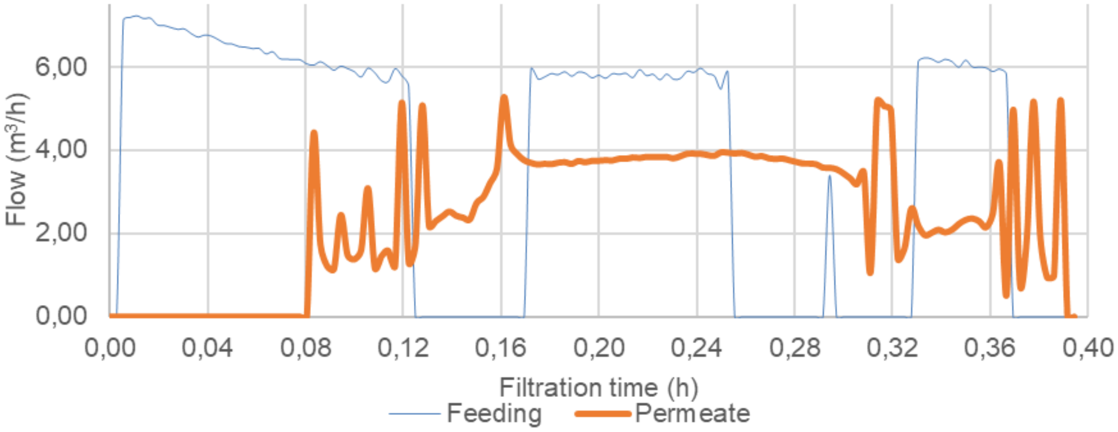

Figure A.52 – Permeability check 03.07.2020.

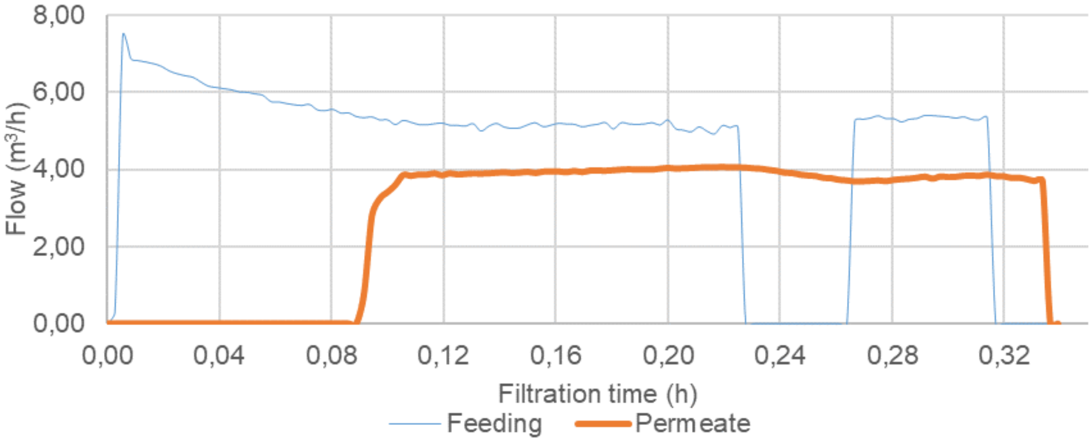

Figure A.53 – Permeability check 03.07.2020a.

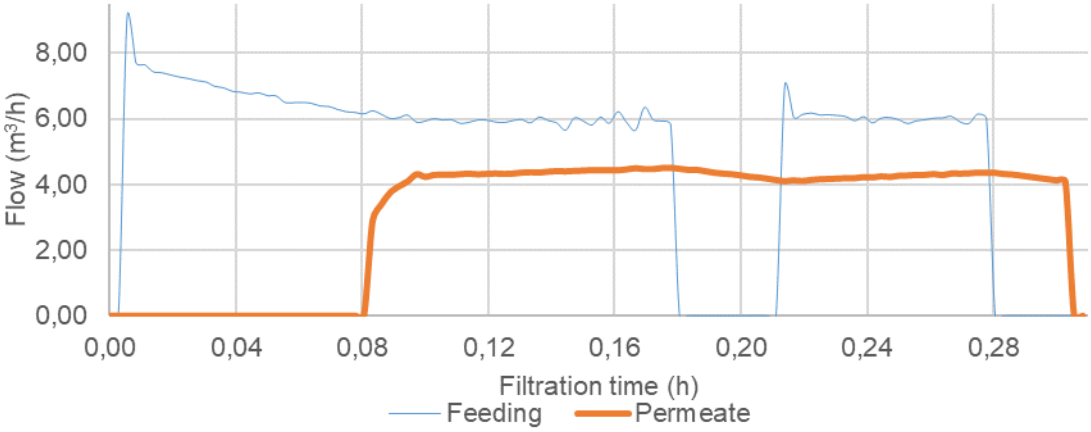

Figure A.54 – Permeability check 03.07.2020b.

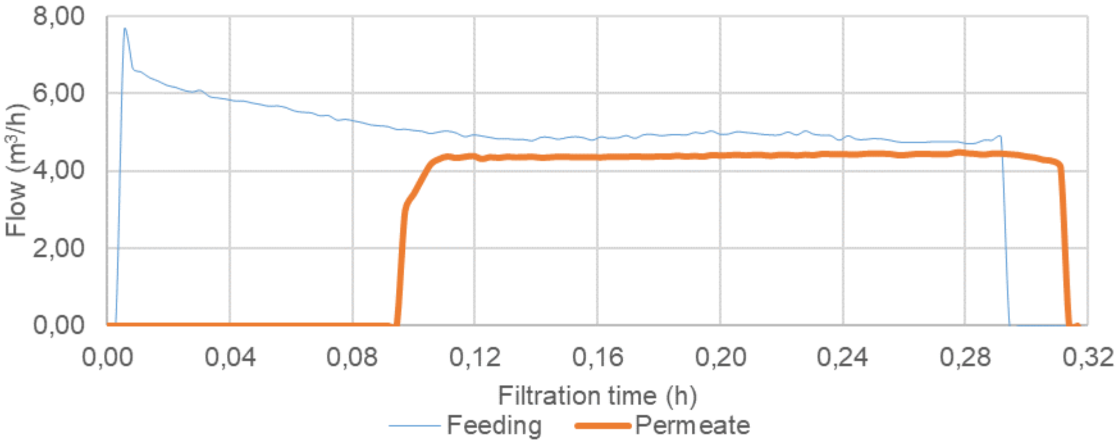

Figure A.55 – Permeability check 03.07.2020c.

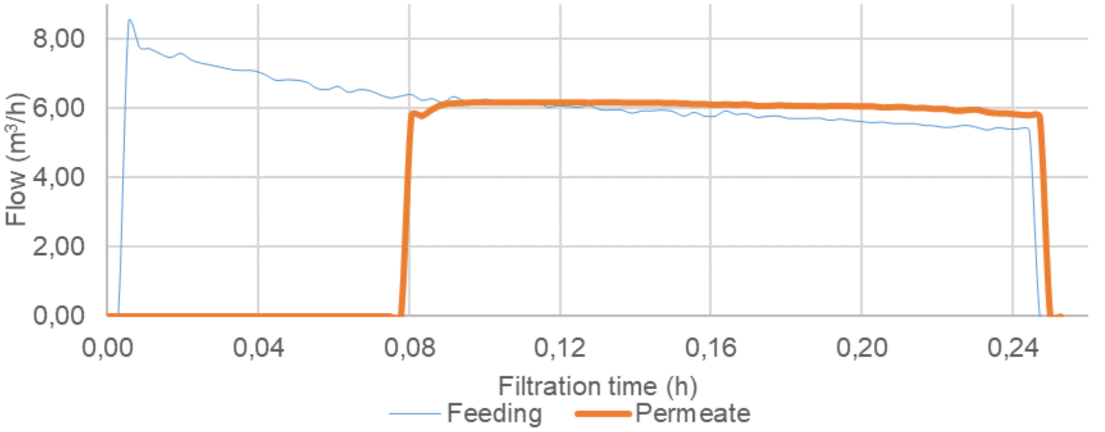

Figure A.56 – Permeability check 10.07.2020.

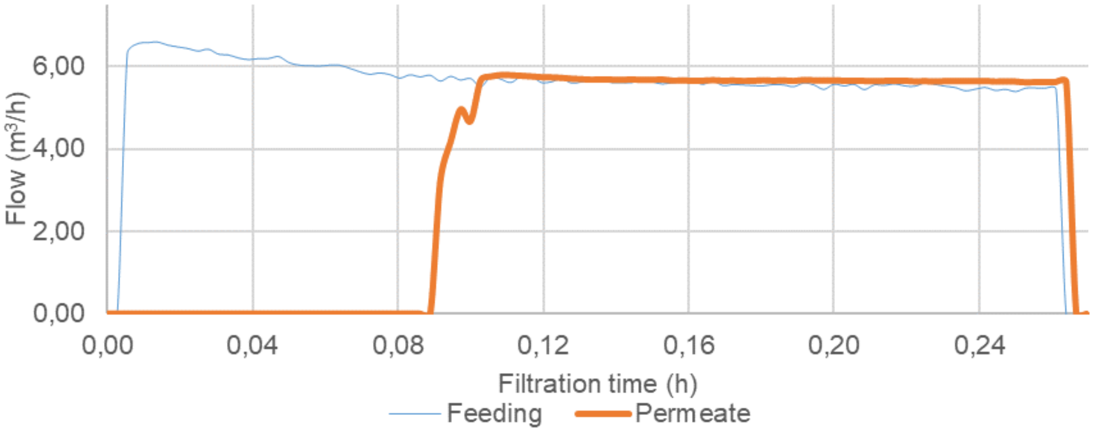

Figure A.57 – Permeability check 10.07.2020a.

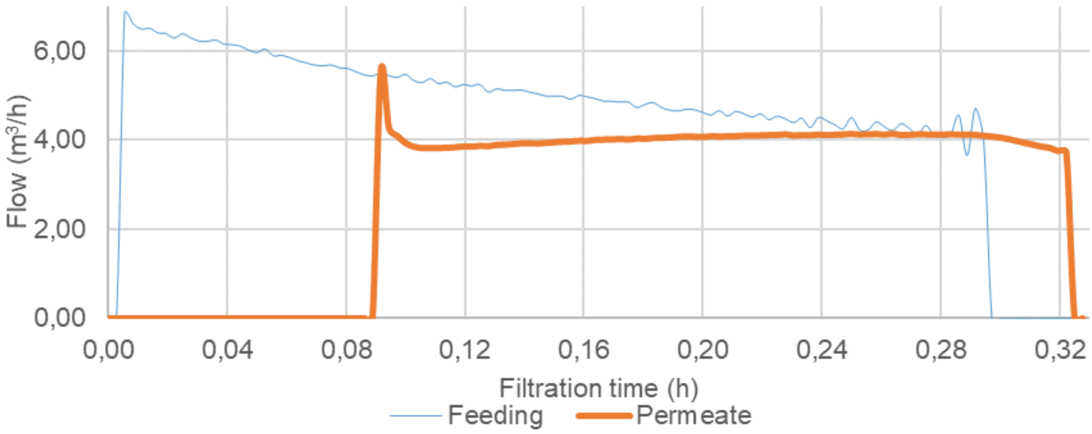

Figure A.58 – Permeability check 10.07.2020b.

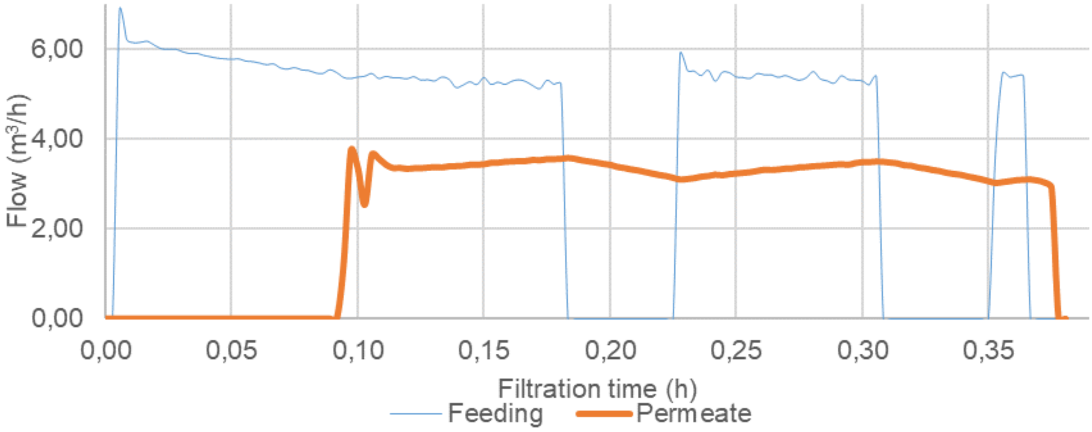

Figure A.59 – Permeability check 13.07.2020.

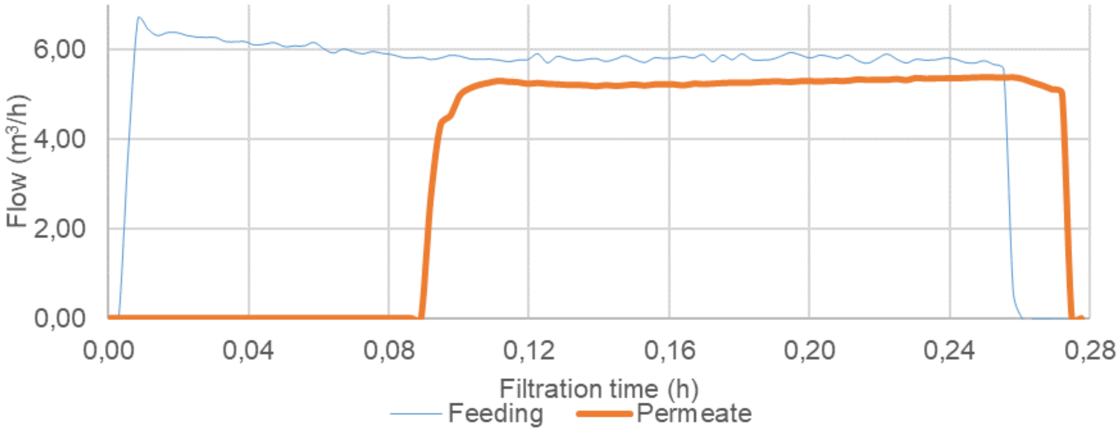

Figure A.60 – Permeability check 28.07.2020.

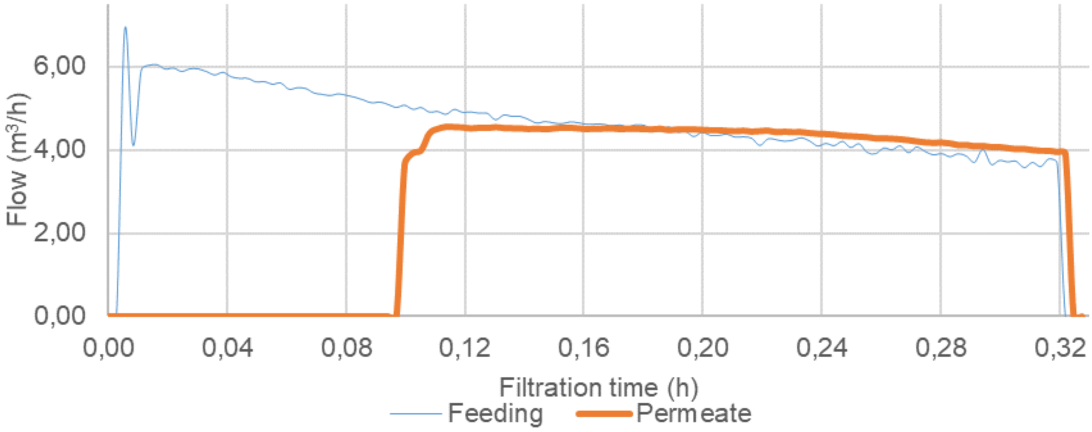

Figure A.61 – Permeability check 28.07.2020a.

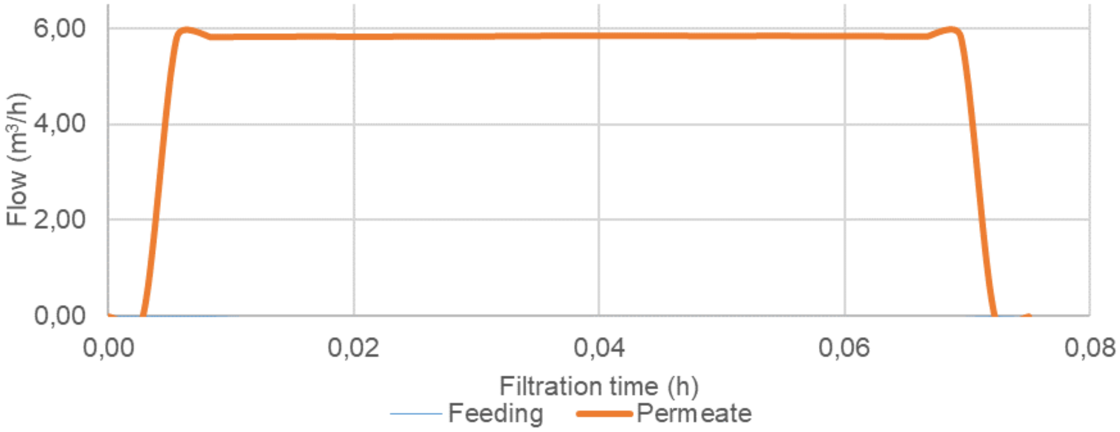

Figure A.62 – Permeability check 29.07.2020.

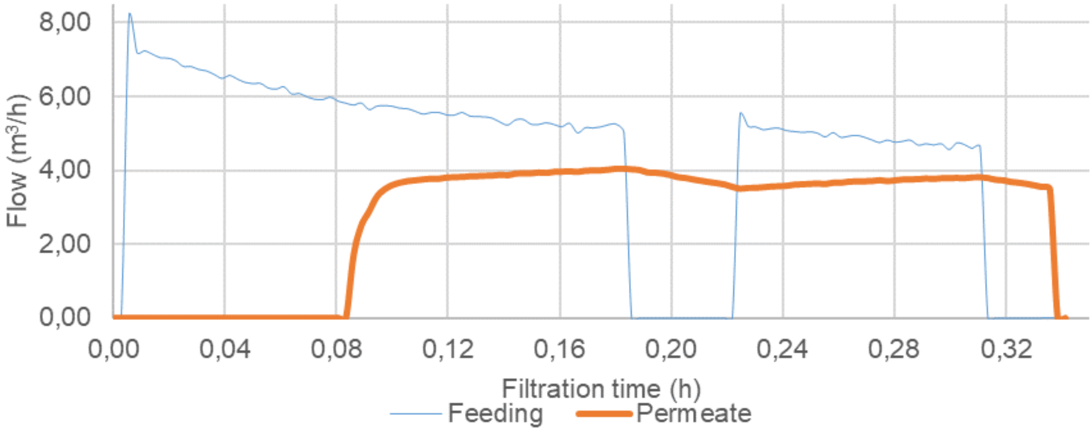

Figure A.63 – Permeability check 29.07.2020a.

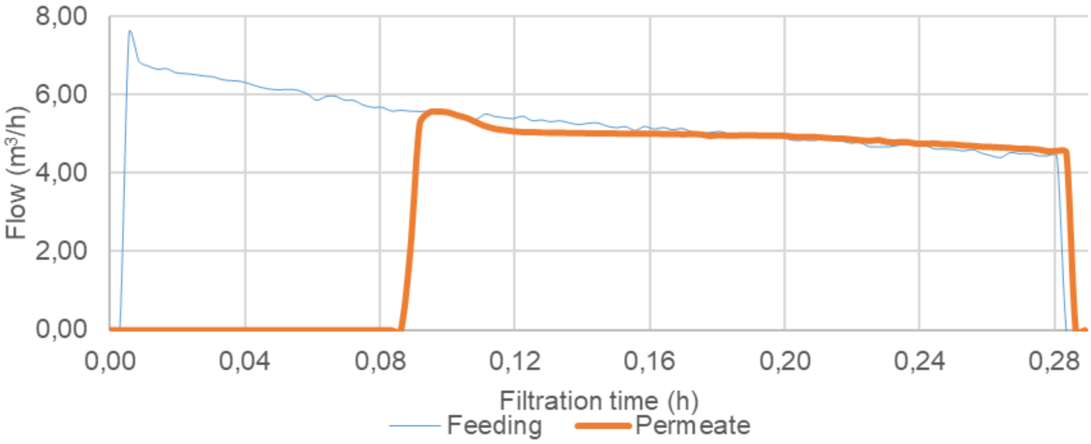

Figure A.64 – Permeability check 30.07.2020.

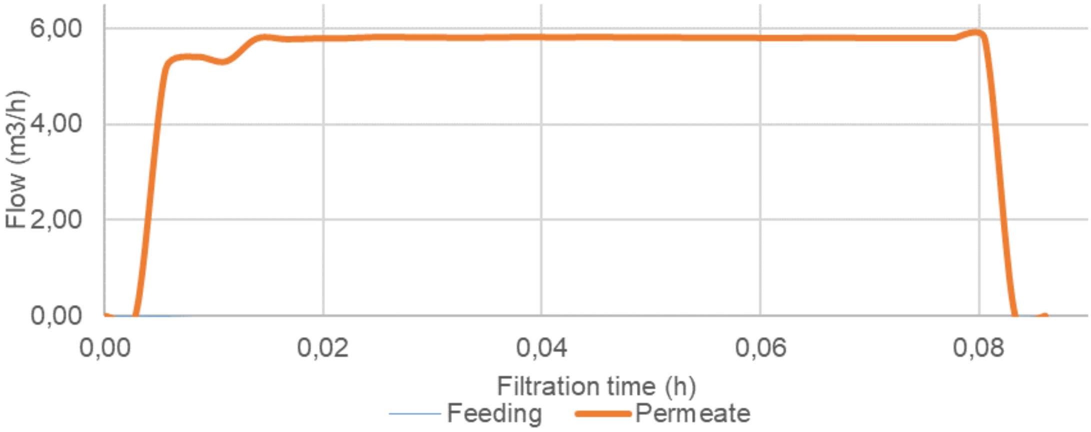

Figure A.65 – Permeability check 30.07.2020a.

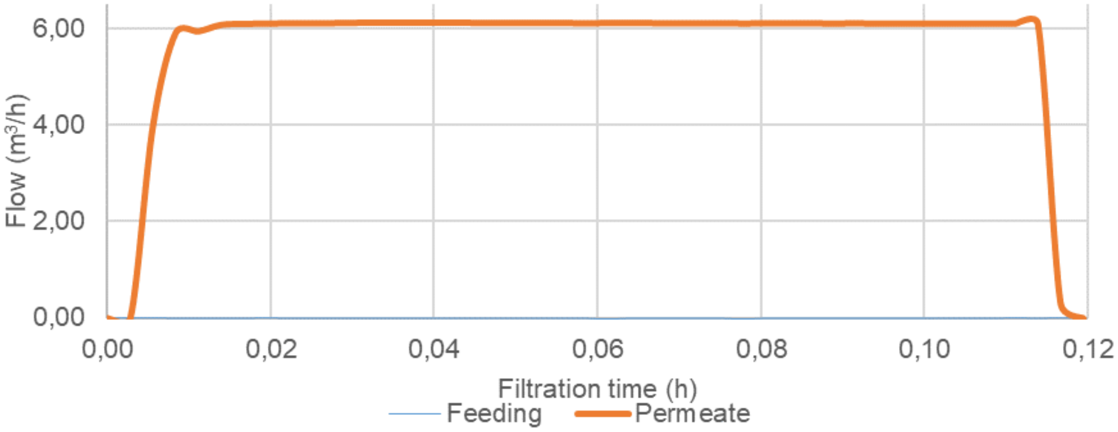

Figure A.66 – Permeability check 12.08.2020.

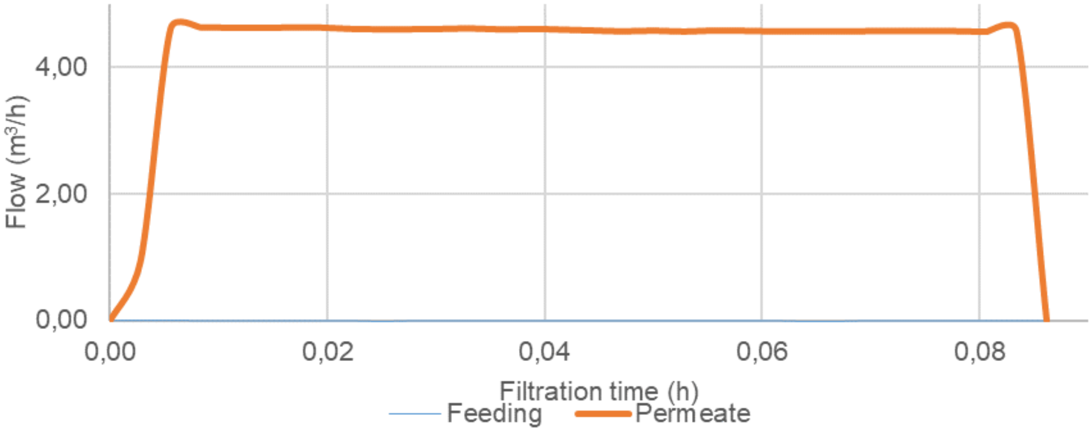

Figure A.67 – Permeability check 12.08.2020a.

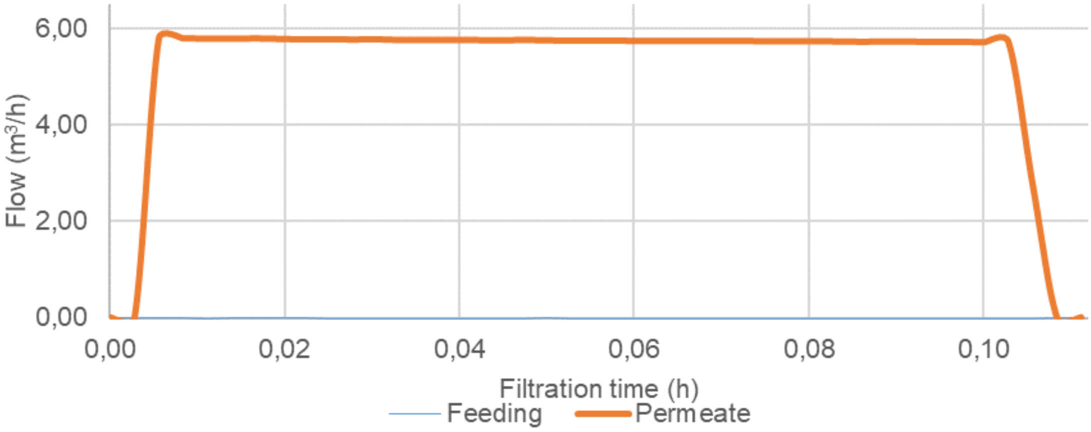

Figure A.68 – Permeability check 13.08.2020.

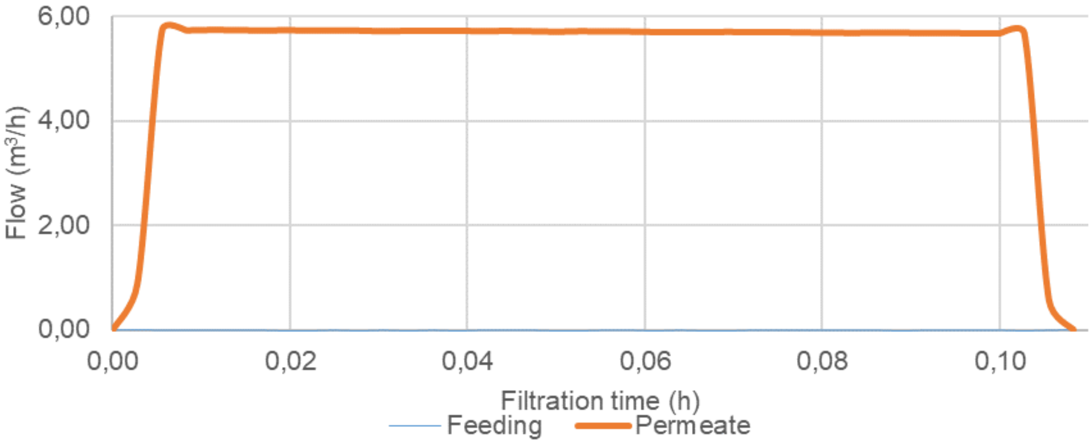

Figure A.69 – Permeability check 13.08.2020a.

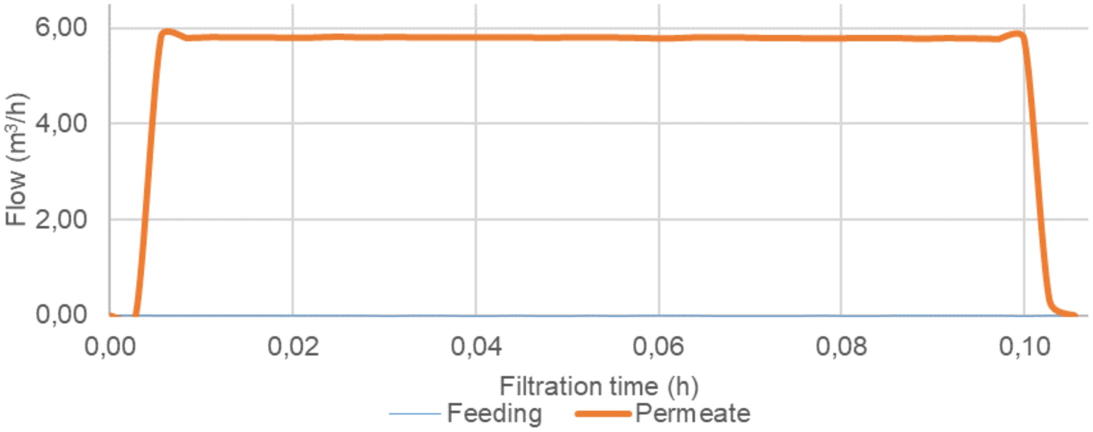

Figure A.70 – Permeability check 13.08.2020b.

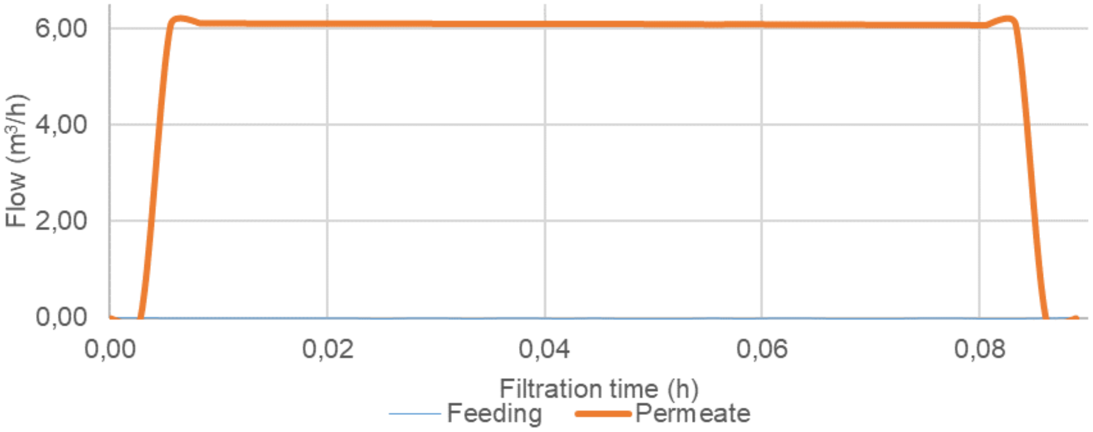

Figure A.71 – Permeability check 17.08.2020.

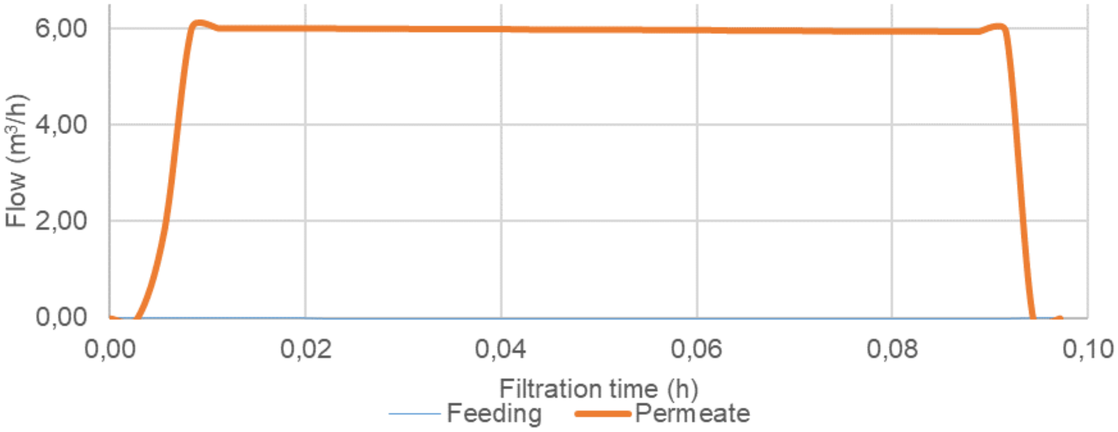

Figure A.72 – Permeability check 17.08.2020a.

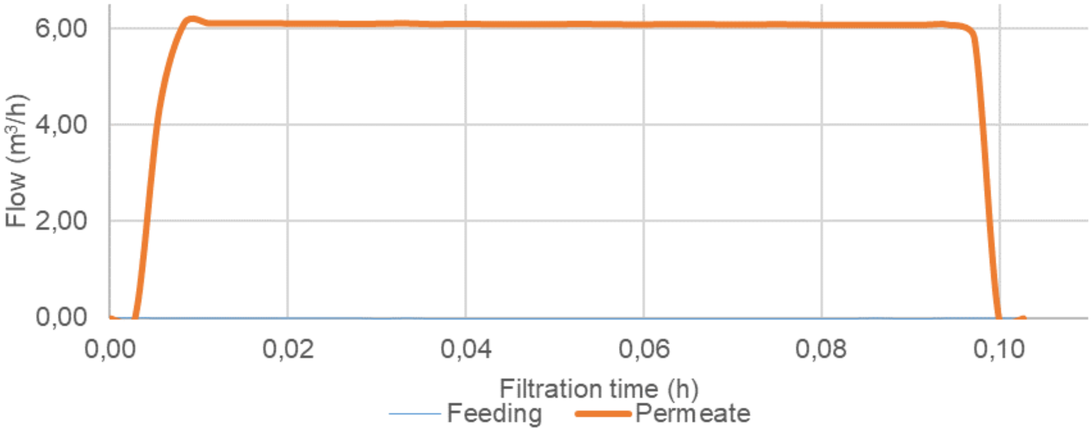

Figure A.73 – Permeability check 19.08.2020.

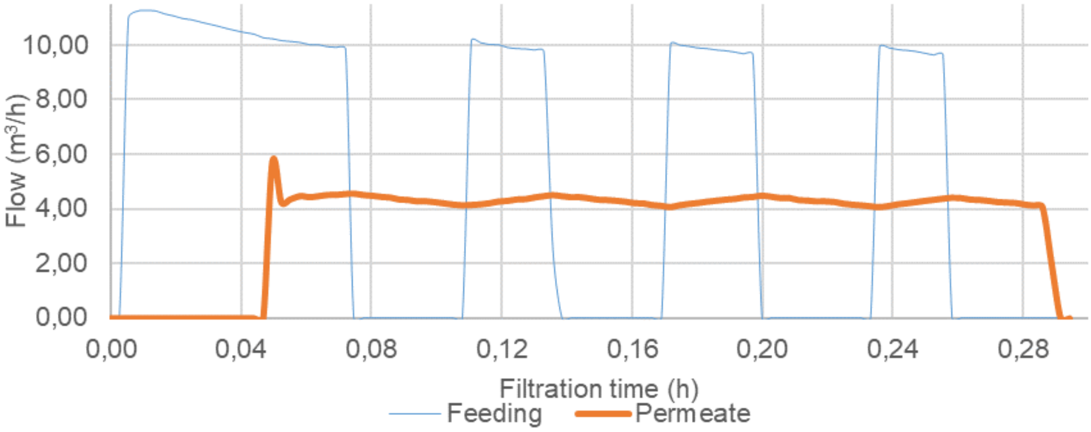

Figure A.74 – Permeability check 19.08.2020a.

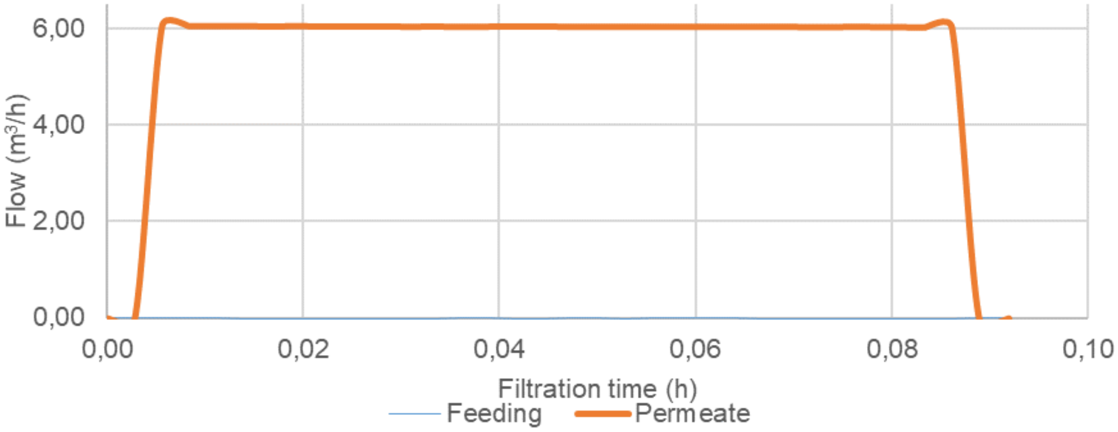

Figure A.75 – Permeability check 20.08.2020.

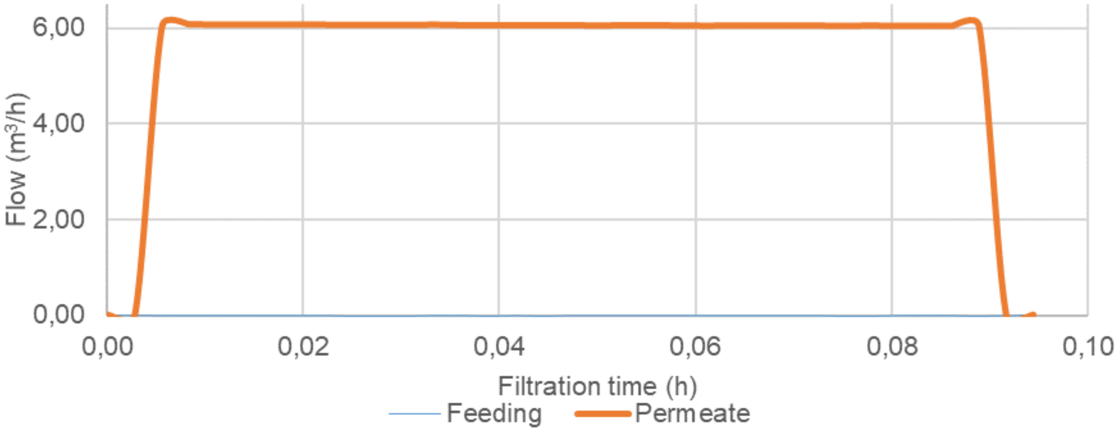

Figure A.76 – Permeability check 21.08.2020.

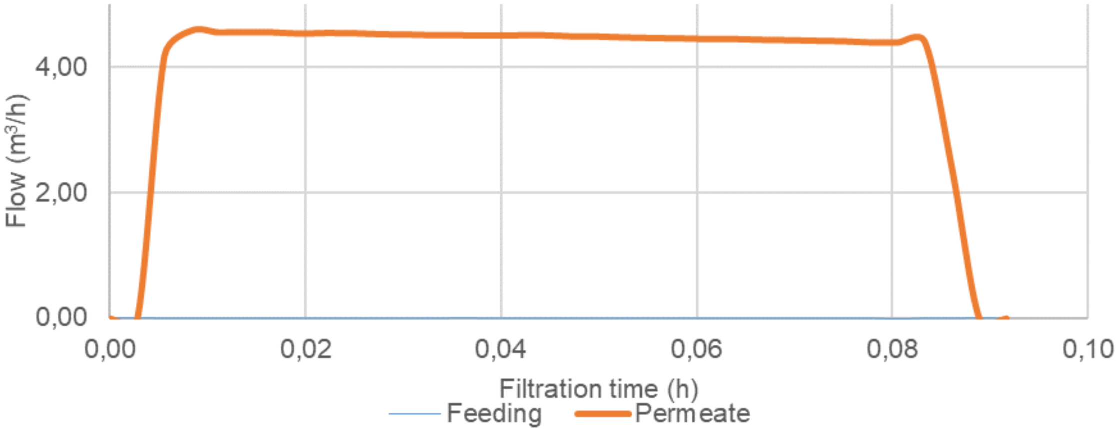

Figure A.77 – Permeability check 21.08.2020a.

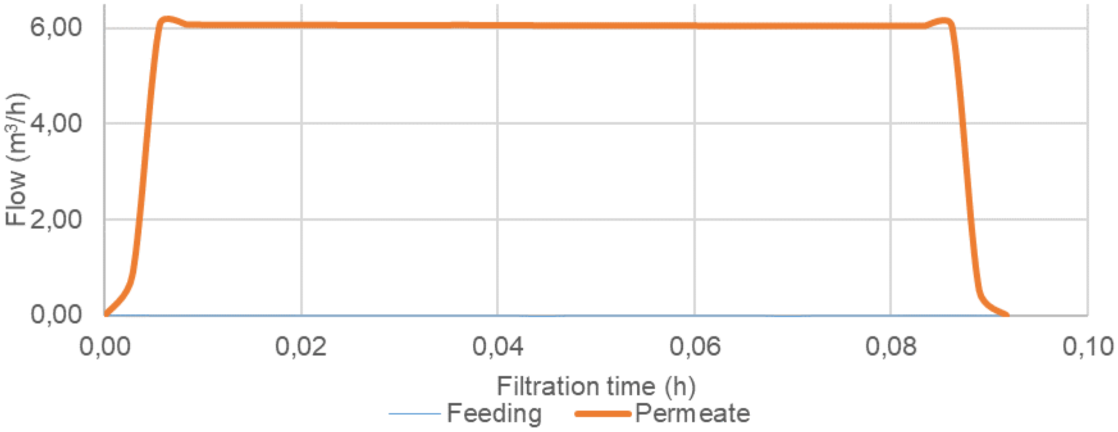

Figure A.78 – Permeability check 24.08.2020.

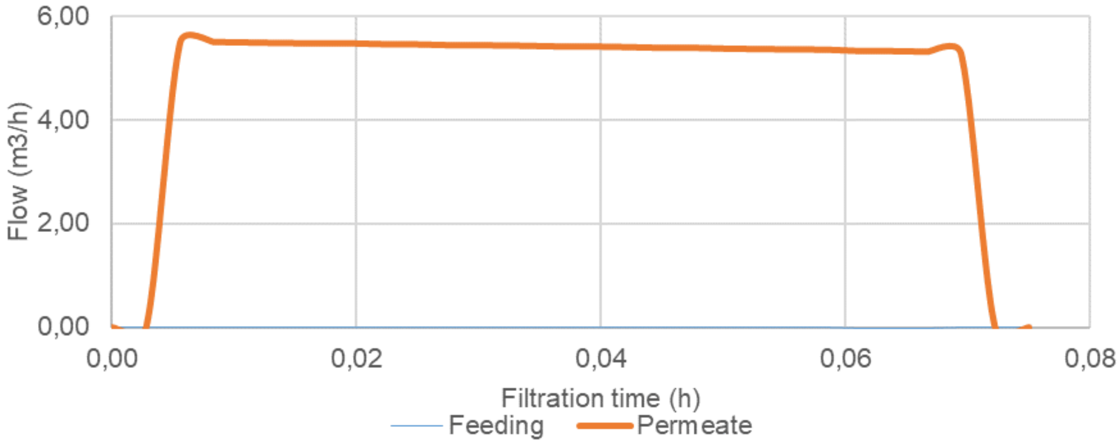

Figure A.79 – Permeability check 24.08.2020a.

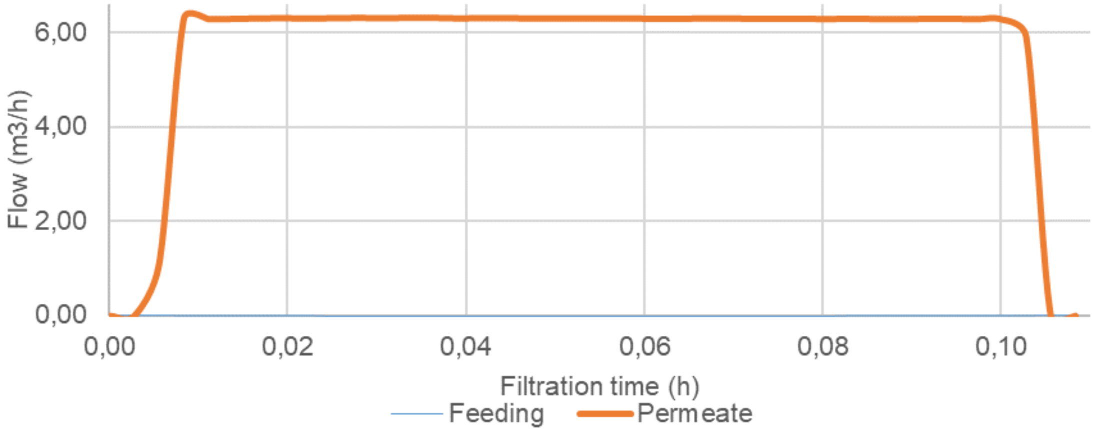

Figure A.80 – Permeability check 03.09.2020.

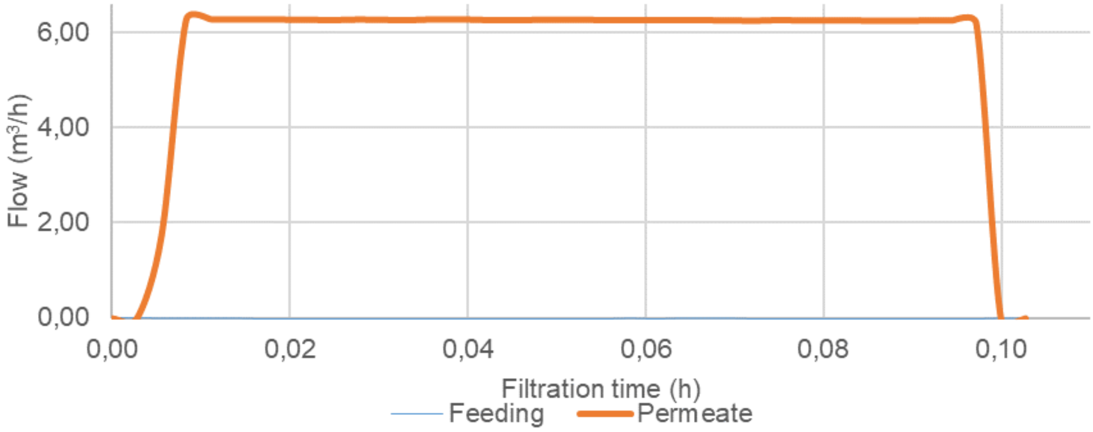

Figure A.81 – Permeability check 07.09.2020.

Appendix B – Nannochloropsis harvest filtration graphics

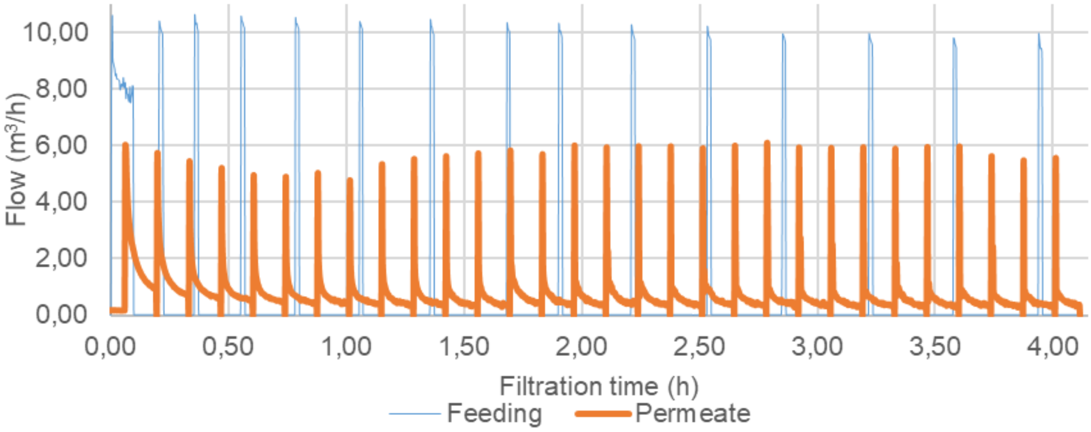

Figure B.82 – Nannochloropsis harvest filtration 02.01.2020.

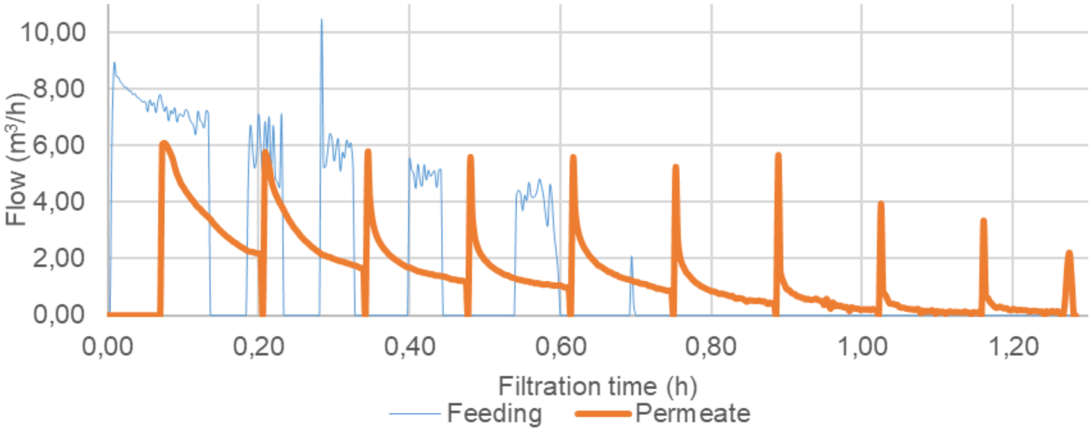

Figure B.83 – Nannochloropsis harvest filtration 23.01.2020.

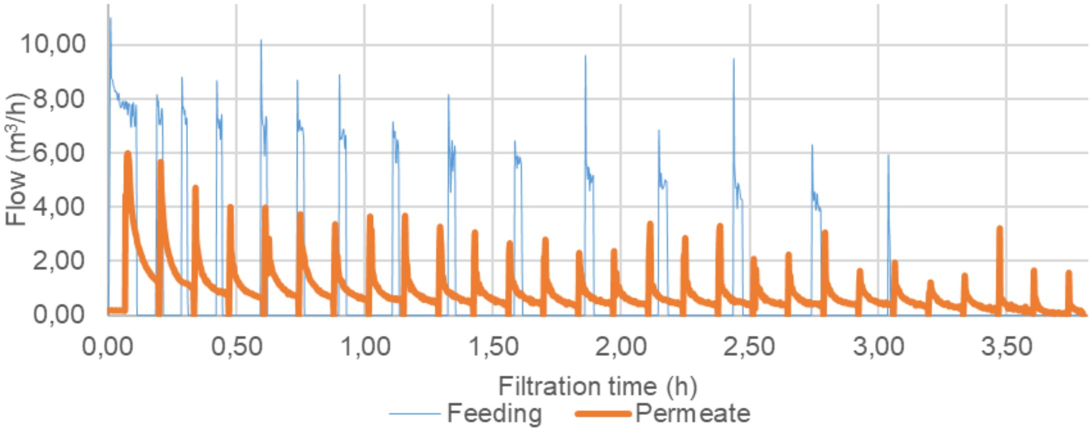

Figure B.84 – Nannochloropsis harvest filtration 28.01.2020.

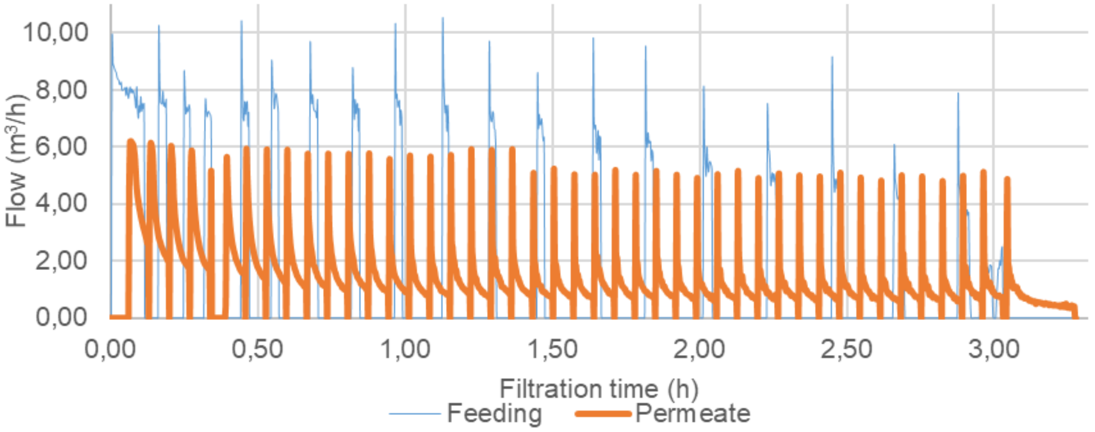

Figure B.85 – Nannochloropsis harvest filtration 11.03.2020.

Appendix C – Odontella harvest filtration graphics

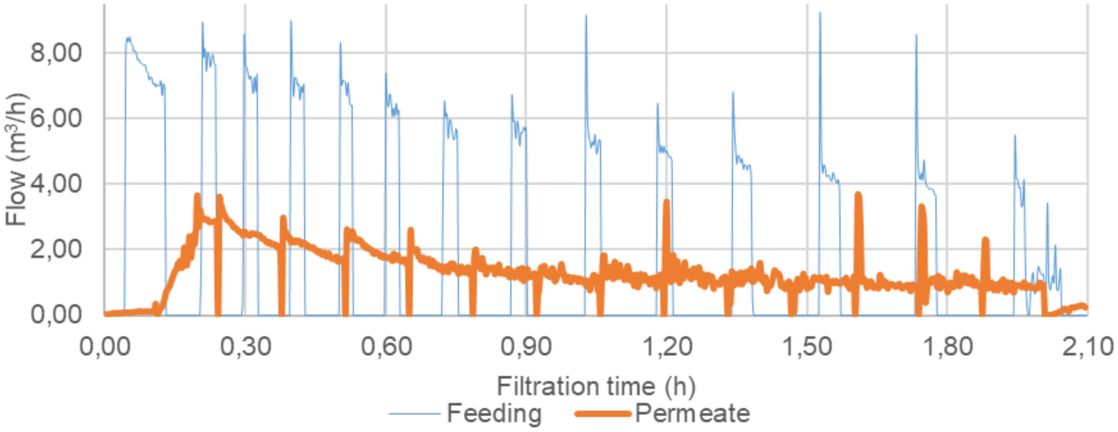

Figure C.86 – Odontella harvest filtration 10.12.2019.

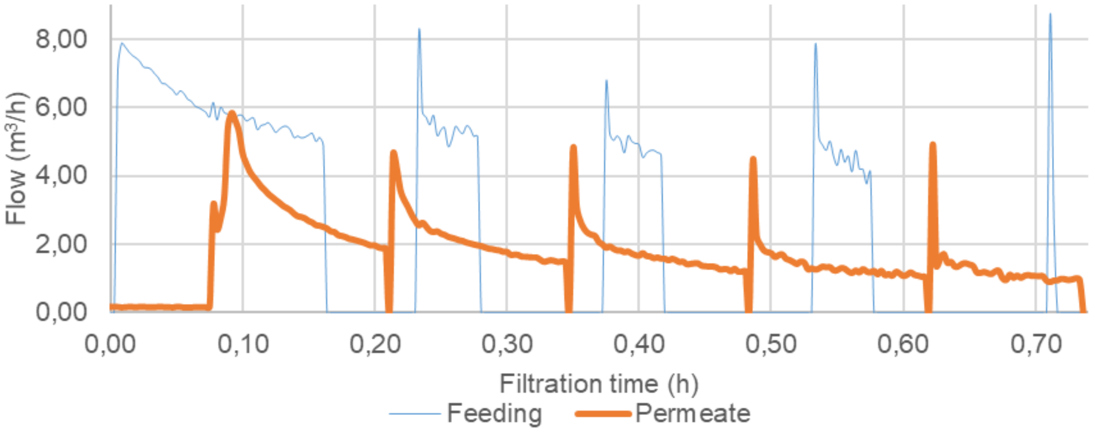

Figure C.87 – Odontella harvest filtration 16.01.2020.

Appendix D – Supernatant *Nannochloropsis* harvest filtration graphics

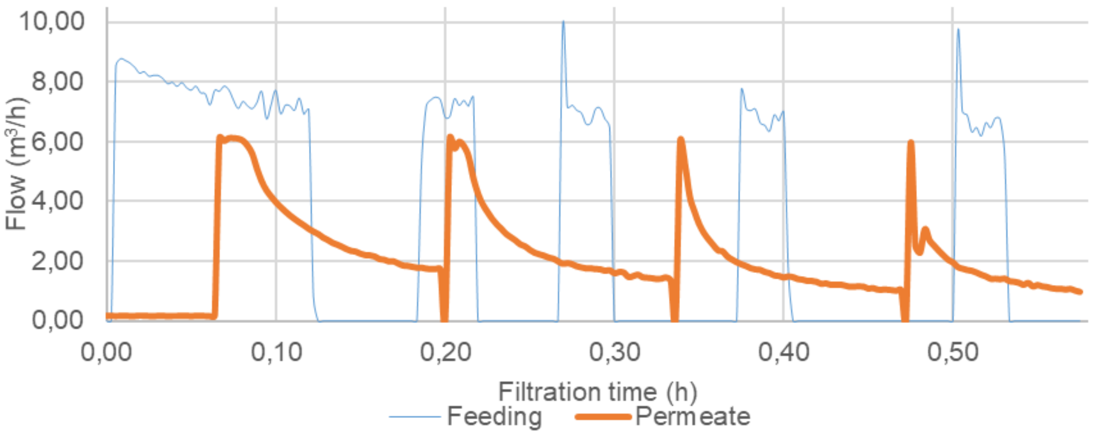

Figure D.88 – Supernatant Nannochloropsis harvest filtration 13.02.2020.

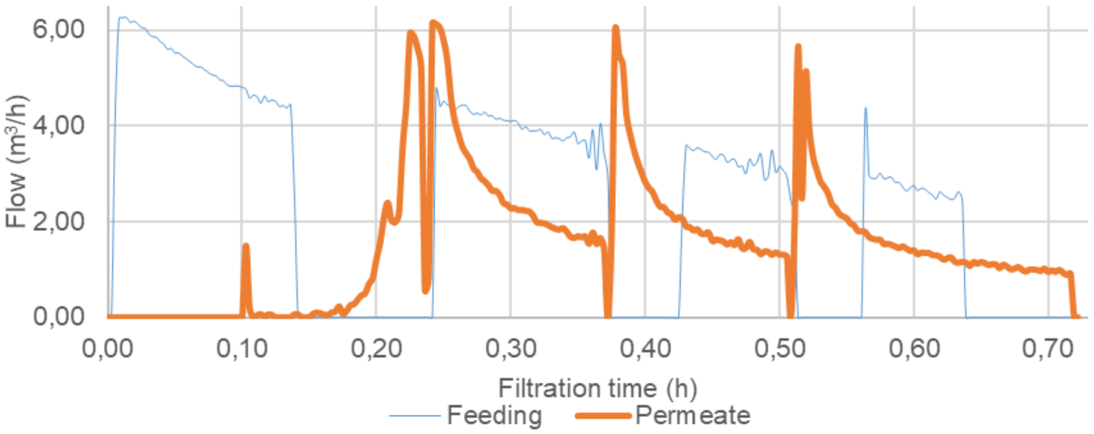

Figure D.89 – Supernatant Nannochloropsis harvest filtration 29.05.2020.

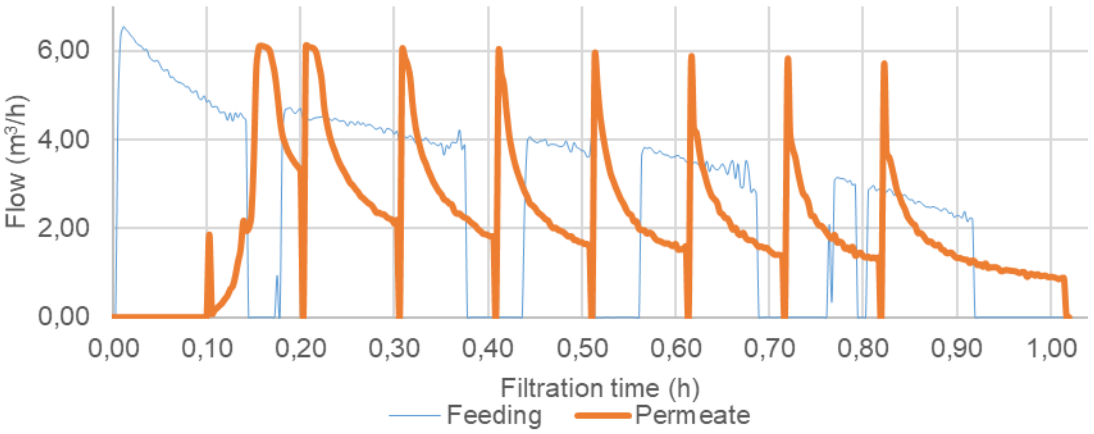

Figure D.90 – Supernatant Nannochloropsis harvest filtration 02.06.2020.

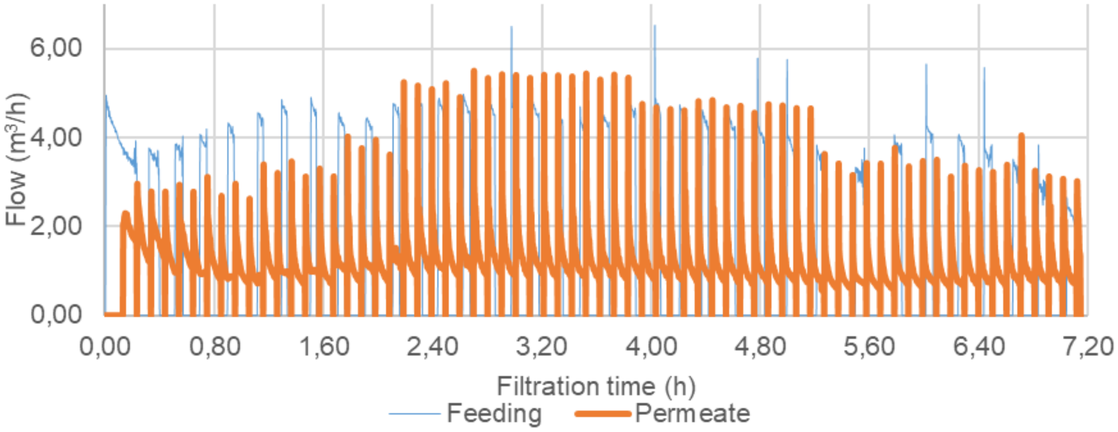

Figure D.91 – Supernatant Nannochloropsis harvest filtration 03.06.2020.

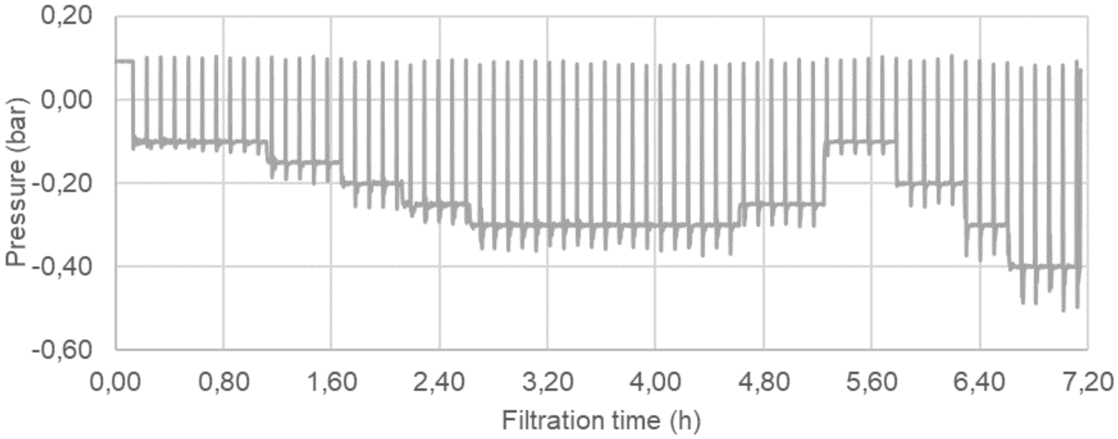

Figure D.92 – TMP in function of the time for the permeability check on Figure D.91.

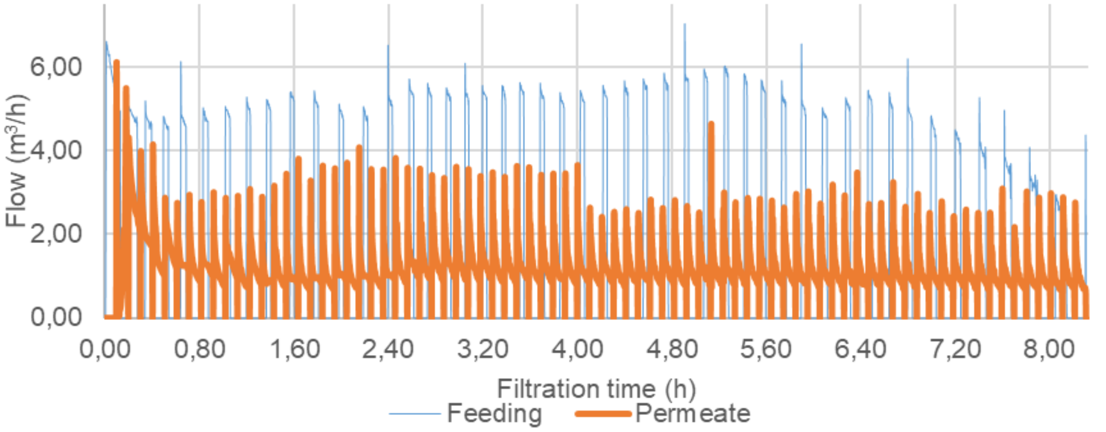

Figure D.93 – Supernatant Nannochloropsis harvest filtration 04.06.2020.

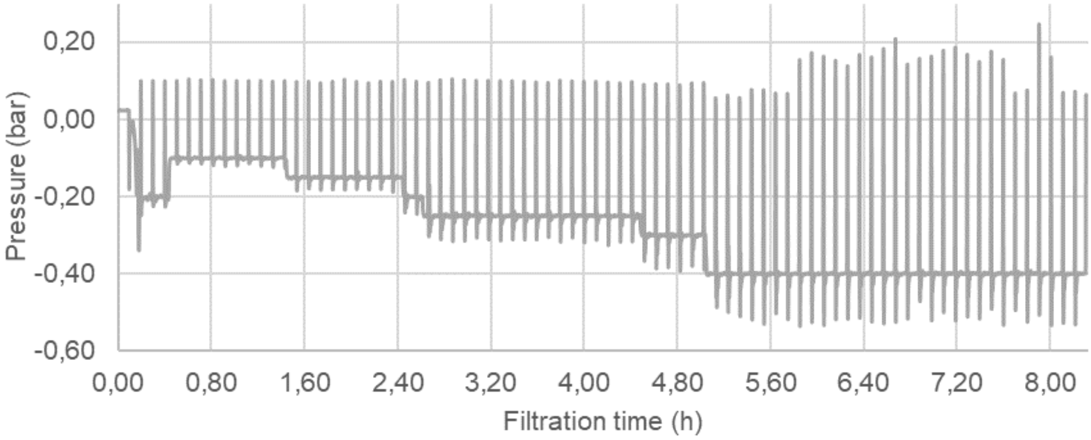

Figure D.94 – TMP in function of the time for the permeability check on Figure D.93.

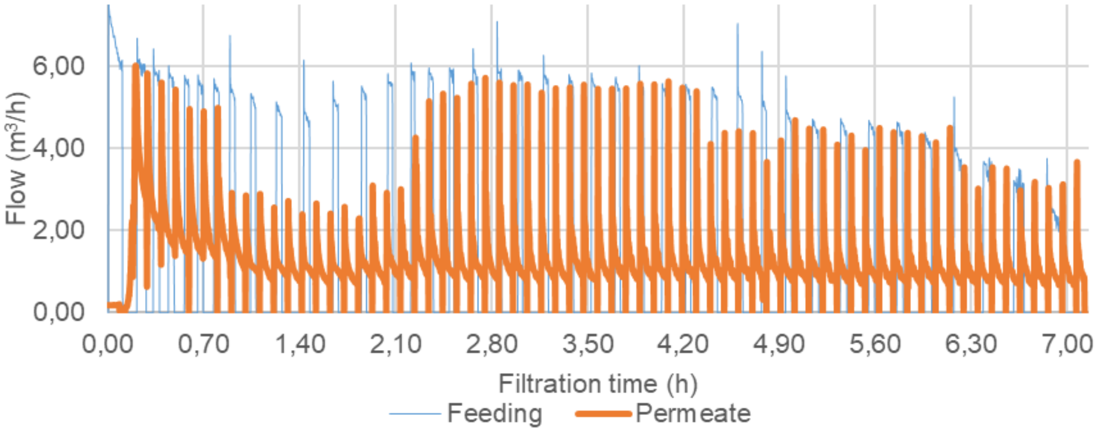

Figure D.95 – Supernatant Nannochloropsis harvest filtration 17.06.2020.

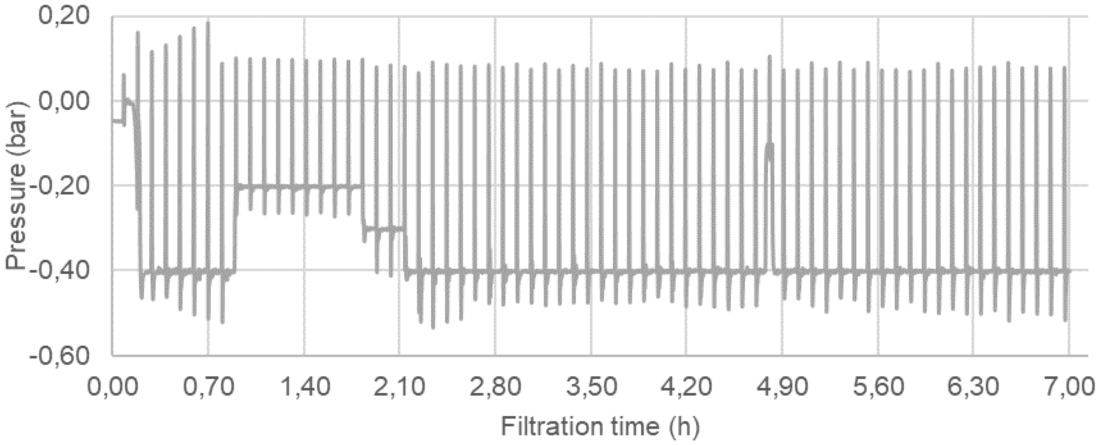

Figure D.96 – TMP in function of the time for the permeability check on Figure D.95.

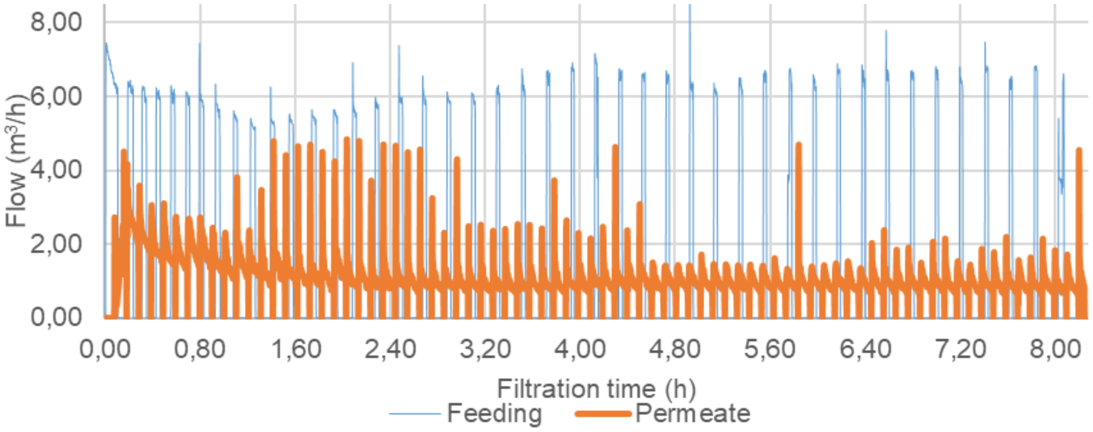

Figure D.97 – Supernatant Nannochloropsis harvest filtration 18.06.2020.

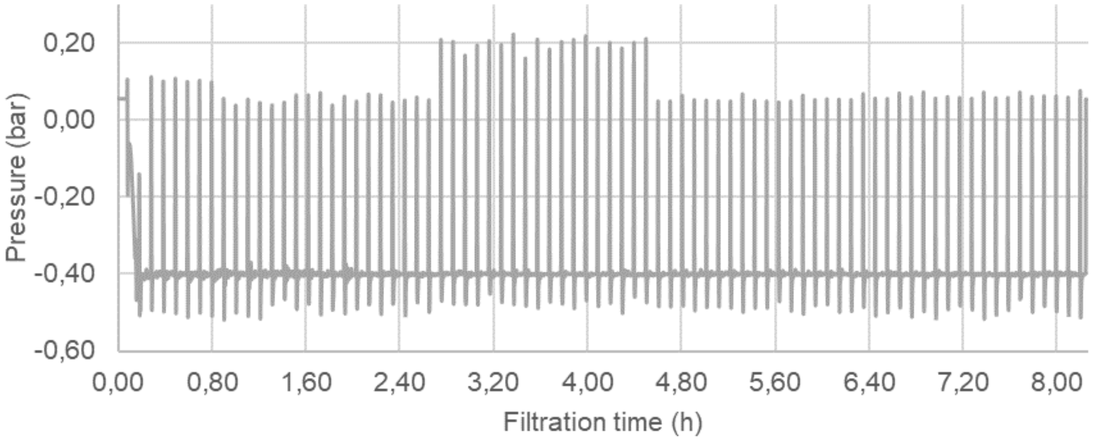

Figure D.98 – TMP in function of the time for the permeability check on Figure D.97.

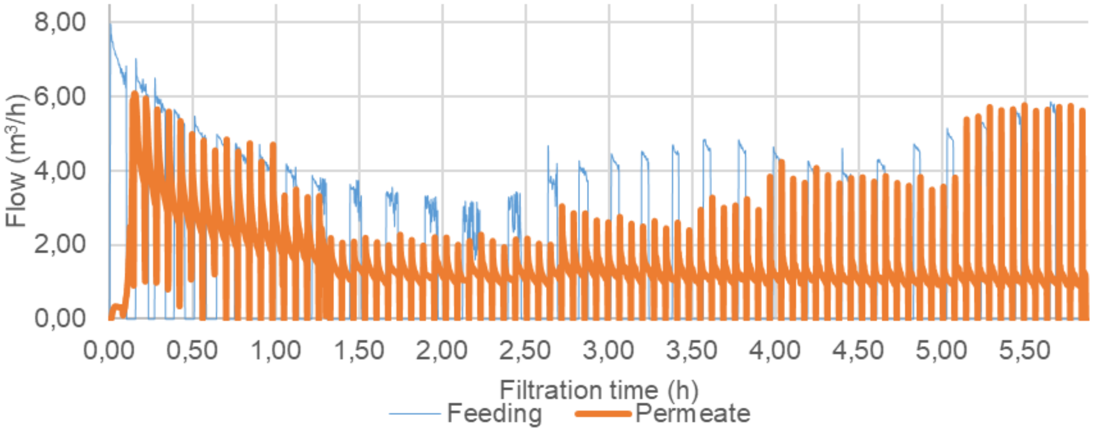

Figure D.99 – Supernatant Nannochloropsis harvest filtration 01.07.2020.

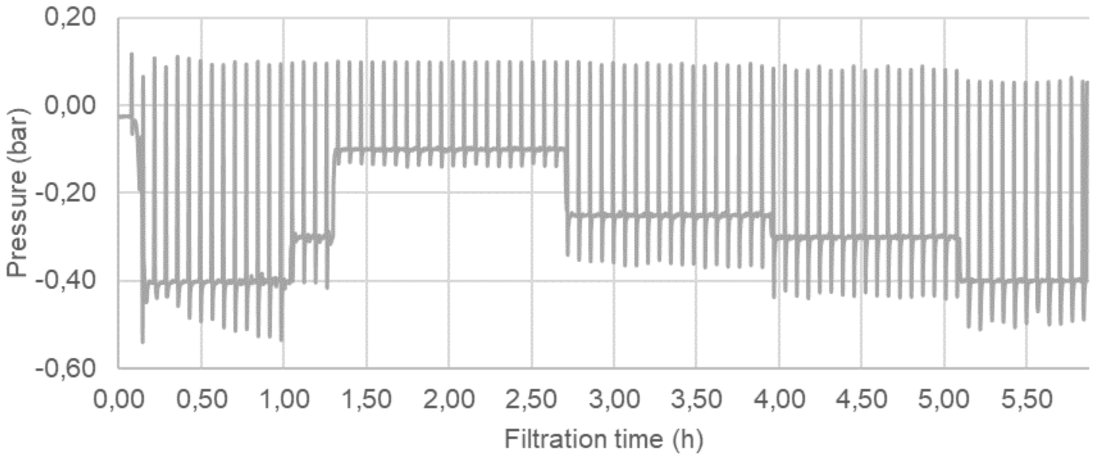

Figure D.100 – TMP in function of the time for the permeability check on Figure D.99.

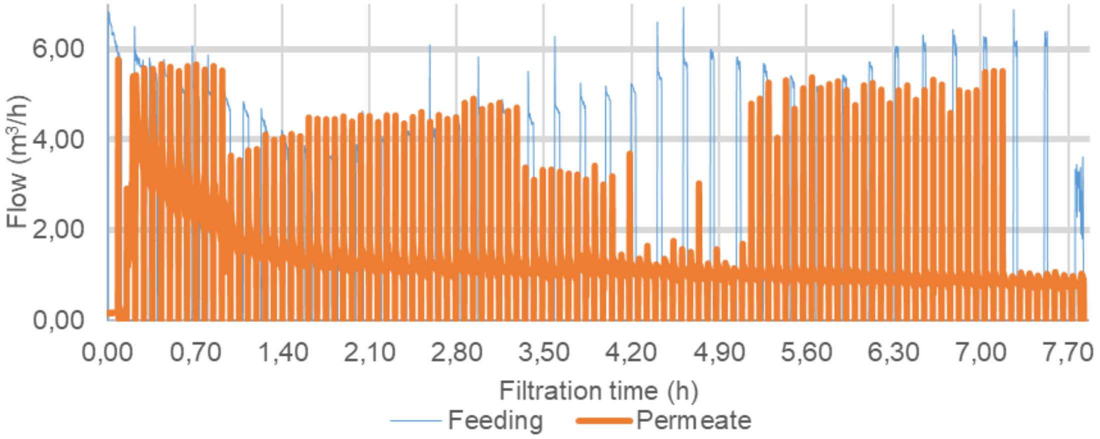

Figure D.101 – Supernatant Nannochloropsis harvest filtration 02.07.2020.

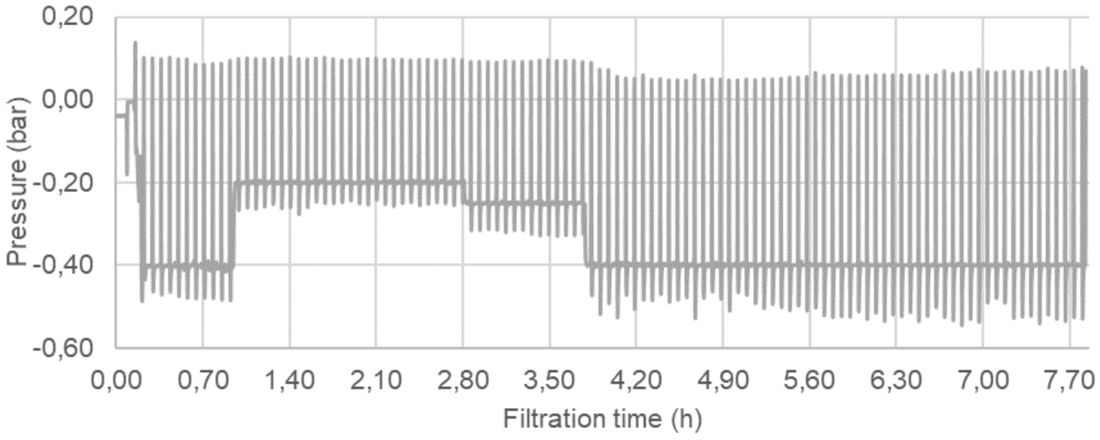

Figure D.102 – TMP in function of the time for the permeability check on Figure D.101.

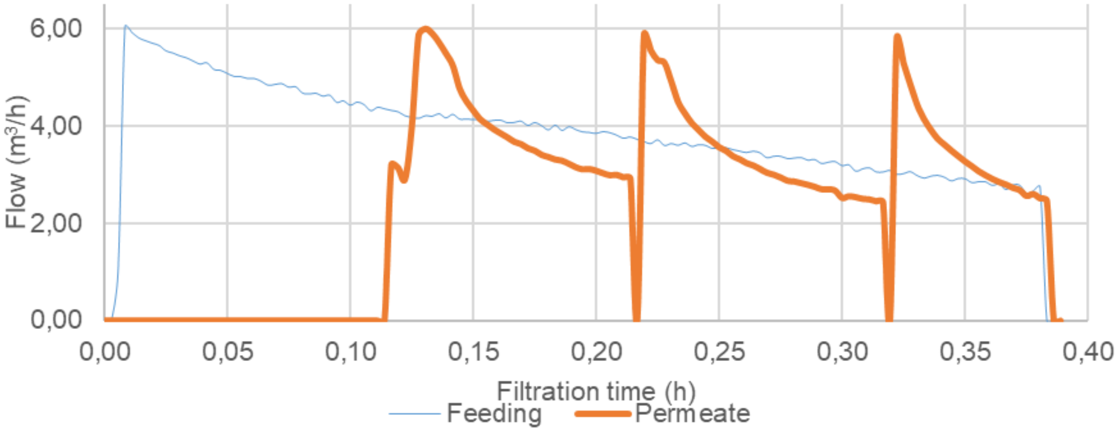

Figure D.103 – Supernatant Nannochloropsis harvest filtration 13.07.2020.

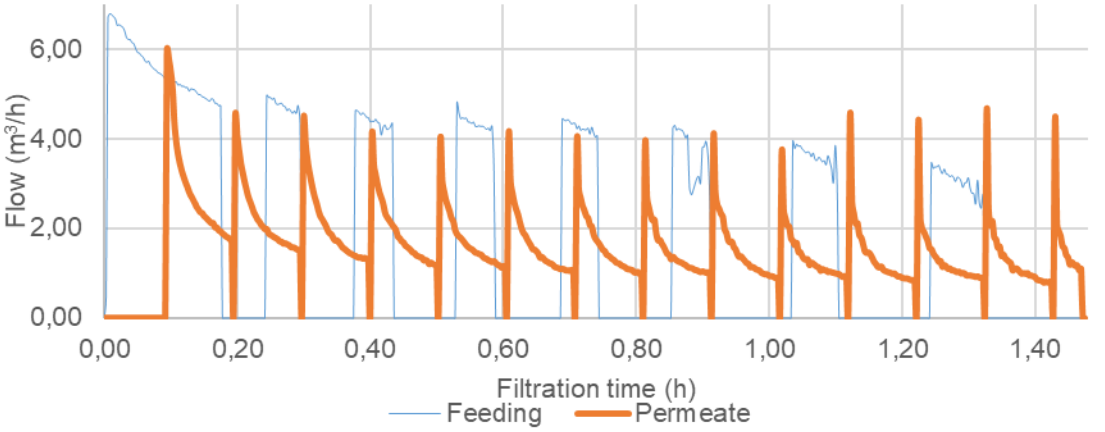

Figure D.104 – Supernatant Nannochloropsis harvest filtration 29.07.2020.

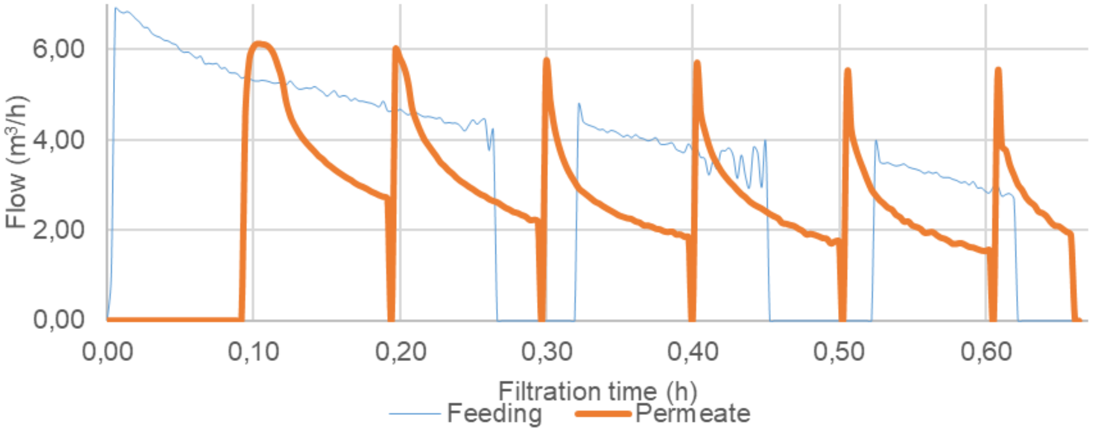

Figure D.105 – Supernatant Nannochloropsis harvest filtration 12.08.2020.

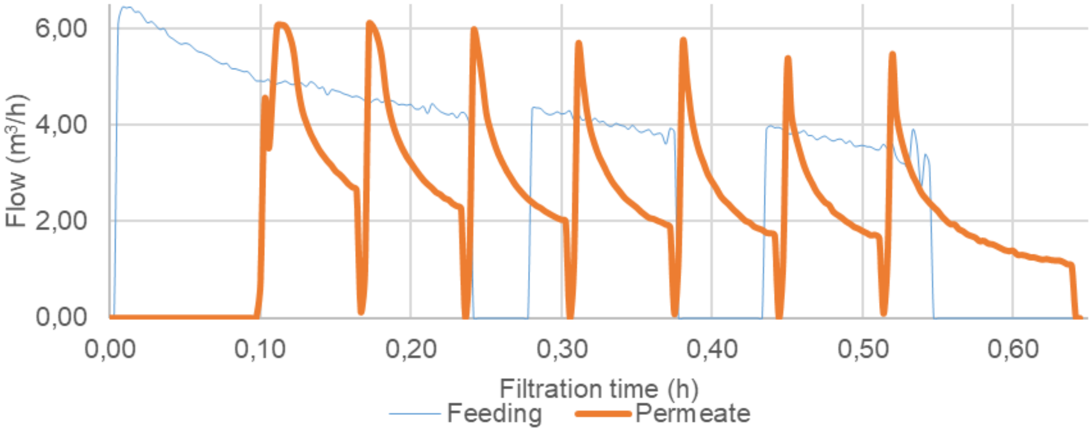

Figure D.106 – Supernatant Nannochloropsis harvest filtration 17.08.2020.

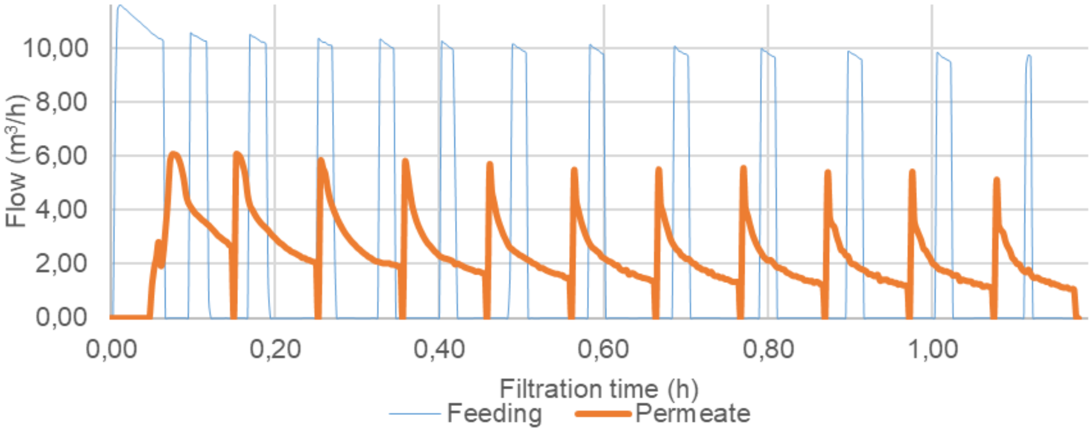

Figure D.107 – Supernatant Nannochloropsis harvest filtration 19.08.2020.

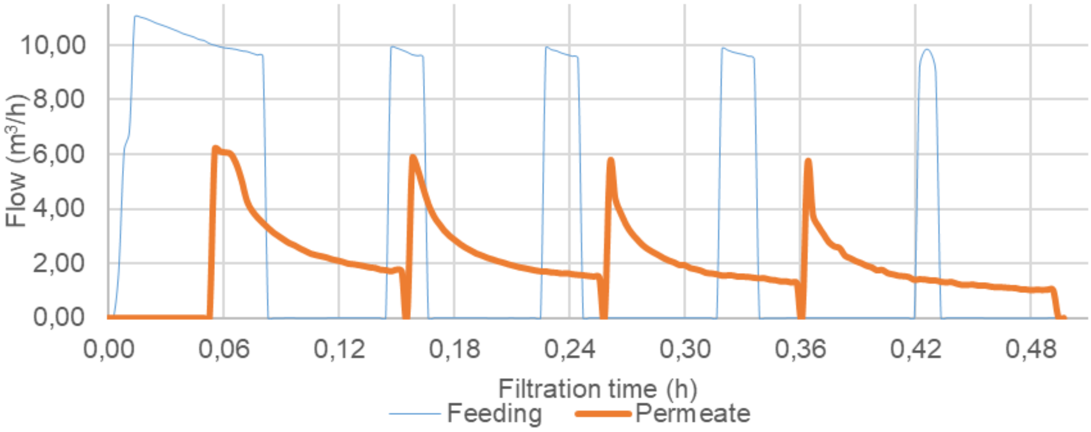

Figure D.108 – Supernatant Nannochloropsis harvest filtration 21.08.2020.

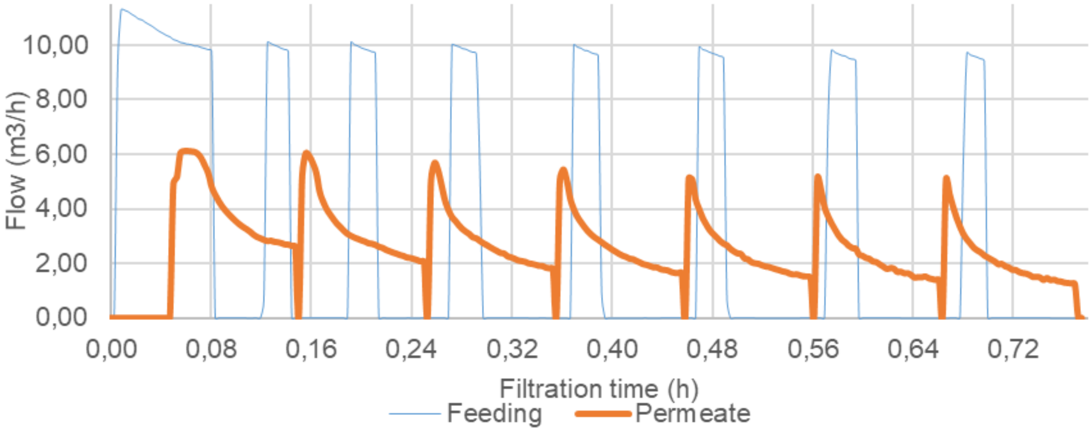

Figure D.109 – Supernatant Nannochloropsis harvest filtration 24.08.2020.

Appendix E – Supernatant *Odontella* harvest filtration graphics

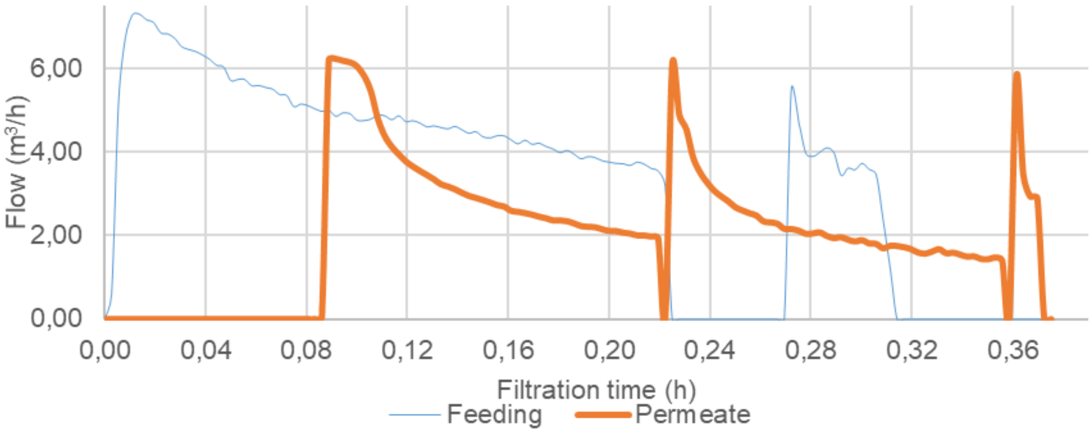

Figure E.110 – Supernatant Odontella harvest filtration 05.03.2020.

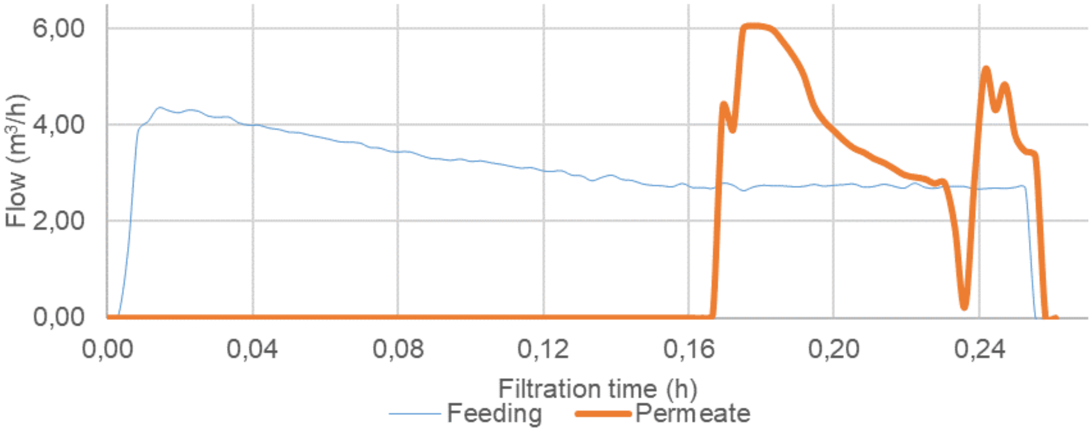

Figure E.111 – Supernatant Odontella harvest filtration 13.08.2020.